# STUDIES ON SOME NONCONVENTIONAL ADSORBENTS FOR THE REMOVAL AND RECOVERY OF HEAVY METALS FROM INDUSTRIAL WASTES

## DISSERTATION

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# Certificate

This is to certify that the work presented in the dissertation entitled, "Studies on some non conventional adsorbents for the removal and recovery of heavy metals from industrial wastes " is the original work of Mr. Moonis Ali Khan, carried out under my supervision and guidance. He has fulfilled all the requirements for the award of M.Phil. degree under Academic Ordinance of the Aligarh Muslim University, Aligarh. The work submitted in this dissertation has not been submitted elsewhere for the award of any other degree or diploma.

(RIFAQAT ALI KHAN RAO)
Supervisor

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MOONIS ALI KHAN

# Contents

# CHAPTER- 1

# Introduction

# Introduction

Natural Environment comprises of three elements air, water and soil. These elements have been provided by the nature, in a beautiful balance and the human life is based on this equation. But, when this balance is disturbed in any way, the phenomenon is termed as Environmental Pollution.

Omnipotent almighty has gifted human beings with the quality to adapt to any kind of circumstances. When life evolved on planet earth, everything was available in its purest form. With the passage of time, the purity started degrading.

Exploitative use of the natural resources by human beings create ecological imbalance. As the pollution level increased, human beings keep on adjusting themselves in the changing scenario. This limit has been termed as the standard of the environment. (M.Rafiq; 1995)

Air is one of the three most important components of natural environment. Fresh air is the primary priority for healthy living. Due to industrial development and other economic processes, proliferation of vehicular traffic and also due to agricultural effluents, air pollution has reached a stage where the public cannot turn blind eye to it any longer. Phenomena like global warming, green house effect, ozone layer depletion and photochemical smog are the threats to the environment of this planet. If not addressed, life would be extinct, with in a very short span of time. The situation is acute and demands urgent action.

Water is the other abundantly available natural resource on the earth surface comprising 70% of the earth surface. Water is essential for sustaining life on our planet. Estimates suggest that nearly 1.5 billion people lack safe drinking water and that at least 5 million deaths per year can be attributed to water borne diseases. Human beings considering these water bodies as a limitless dumping ground for wastes. Raw sewage, garbage and oil spills have begun to overwhelm the diluting capabilities of the oceans, and most costal waters are now polluted. The way human activities immensely polluting these natural resources is like they are digging their own graves.

Soil is an other element of life, as the life initiated from soil and the forces, which are sustaining life, are also dependent on soil. Today, the earth is facing many sorts of environmental threats, soil erosion, misuse and unwise use of chemicals and fertilizers are the few. Soil erosion is caused due to disappearance of the forests from the earth. The other source of threat to soil is the misuse of land. The wrong irrigation of land is the major cause of water logging and salinity. The unsystematic use of fertilizers is another threat to the land. Along with the loss of land fertility, the chemicals in the fertilizers go deep into sub soil water, which ultimately play havoc with the health of the human beings

Science provides many practical solutions to minimize the present level at which pollutants are introduced into the environment and for remediating past problems.

Identification and characterization of the pollutants is as important as control of pollution. As after getting relevant information about the pollutant, it is much easier to opt the exact process to control pollution. Estimation and characterization of toxic materials often at trace level in a very complex matrix is a challenging task. Organic as well as inorganic pollutants are not only toxic but also often carcinogenic and affect physiological processes, in such a way that there are long run consequences, which are usually fatal. Moreover, complex organic mixtures are much more difficult to separate and more difficult to identify. To overcome these problems high performance liquid chromatography (HPLC) is playing an exclusive and notable role as one of the most powerful methods of separation. The analysis of inorganic compounds present in water is possible by atomic absorption spectrophotometer (AAS), X-ray fluorescence etc.

However, the greater need of the day is to develop field methods as they have wider applicability and greater flexibility.

Chromatography and ion exchange techniques are the most amenable to such approach. Synthetic inorganic ion exchangers can be used for separation of heavy metals in industrial wastes. Another way is to study the selective adsorption of the toxic species on the exchanger since exchange and adsorption usually go together. However, inorganic ion exchangers are not only expensive to prepare but are also irreproducible, characterized by low capacities and questionable selectivities.

Therefore the use of non-conventional adsorbents is an economically feasible application.

## 1.1: Classification of pollution

Environmental Pollution in general may be classified as:

1.Air Pollution
2.Water Pollution
3.Soil Pollution

**1. Air Pollution**: Many of the processes involved in the industry, commerce, agriculture and in home produce air pollution. Specific examples are the combustion processes used to provide heat and electricity and to power transport, the manufacture of refrigerants and solvents by industry and the decomposing matter on the landfill sites.

At normal levels, air pollution will not cause any serious short-term effects if human health is good, although if levels become very high it may find eyes feel irritated and have a slight cough. At high concentration air pollutants can damage airways to the lungs, causing inflammation and breathing difficulties particularly for individuals with asthma or chest complaints.

Air pollution can be minimized by using high chimneys in industrial plants, so that smoke, fumes or heated air may not disperse in lower atmosphere. The fuels used to run automobiles should be free from air pollutants. Organic wastes should be treated properly. Finally the most effective way to overcome the problem of air pollution is the development of green areas, as green belt development serves as a sink for air pollutants.

**2. Soil Pollution**: Domestic, municipal, industrial, and agricultural wastes are the main source of soil pollution. With advancing agro technology, use of huge quantities of fertilizers, pesticides, herbicides, weedicides, and different soil conditioning agents that are used to increase the crop yield may also contaminate the soil. Synthetic chemicals and fertilizers are the source of trace metals that are added to the soil either deliberately or as an impurity. Radioactive waste containing radio nuclides such as Sr-90, I-129, and Cs-137 are most injurious. Sr-90 gets deposited in bones and tissues instead of Ca. domestic waste including papers, plastics and many notorious chemicals. Other items like paints and varnishes, which are used to add color and gloss to every day life, also add poison to soil. Soil gets large quantities of human, animals and birds excreta, which constitute the major source of land pollution by biological agents. Apart from this faulty sanitation, municipal garbage, wastewater and wrong methods of agricultural practices induce heavy soil pollution.

3. **Water Pollution**: Water is said to be polluted, if its physical and chemical properties are altered due to the addition of large amounts of waste materials, which makes it unfit for its intended use.

Water pollution can be classified as point source and non point source. Point source of pollution occurs when harmful substances are emitted directly into a body of water. The Exxon Valdez oil spill best illustrates point source water pollution. A non point source delivers pollutants indirectly through environmental changes. An example of this type of water pollution is when fertilizer from a field is carried into a steam by rain, in the form of run off which in turn effects aquatic life. The technology exists for point sources are much more difficult to control. Pollution arising from non point sources accounts for a majority of the contaminants in streams and lakes.

## 1.2: Classification of Water Pollutants

Water pollutants can be broadly classified into the following five major categories:

1.Organic Pollutant

2.Inorganic Pollutant

3.Suspended solids and Sediments

4.Radioactive material

1.**Organic Pollutants**: The organic pollutants include domestic and animal sewage, bio-degradable organic compounds and industrial wastes from food processing plants, meat-packing plants, slaughter-houses, paper and pulp mills, tanneries, pathogenic microorganisms, pesticides, detergents, insecticides, paints, sewage, agricultural run off, oil spills from cargo oil tankers on the seas, etc.

These pollutants result in rapid depletion of dissolved oxygen (D.O.) from the water, which is harmful for aquatic organisms. The optimum D.O. in natural waters is 4-6 ppm, which is essential for supporting aquatic life. Any decrease in this D.O. value is an index of pollution. Many aquatic organisms cannot survive at lower D.O. levels in water. The pathogenic microorganisms present in polluted water causes water-born diseases such as cholera, typhoid, dysentery, polio and hepatitis in humans.

The pesticides, detergents, insecticides, paints and other industrial chemicals are toxic to plants, animals and humans, as these chemicals may enter the hydrosphere either by spillage during transport and use or by intentional or accidental release of wastes from their manufacturing establishments.

Oil pollution results in the reduction of light transmission through surface waters, thereby reducing photosynthesis by marine plants. Further, it reduces the D.O. in water and endangers water birds, costal plants and animals. Thus, oil pollution leads to unsightly and hazardous conditions, which are deleterious to marine life and seafood.

**2. <u>Inorganic Pollutants</u>**: Inorganic pollutants comprises of mineral acids, inorganic salts, finely divided metals or metal compounds, trace elements, cyanides, sulphates, nitrates, organometallic compounds and complexes of metals with organics present in natural waters. The metal-organic interactions involve natural organic species. These interactions are influenced by or influence redox equilibria; acid-base reactions, colloid formation and reaction involving microorganisms in water and metal toxicity in aquatic ecosystems are also influenced by these interactions.

Various metals and metallic compounds released from anthropogenic activities add up to their natural background levels in water. Some of these trace metals play essential roles in biological processes, but at higher concentrations, they may be toxic to biota

**3. <u>Suspended solids and sediments</u>**: Sediments are mostly contributed by soil erosion by natural processes, agricultural development, strip mining and construction activities, Suspended solids in water mainly comprise of silt, sand and minerals eroded from the land. Soil erosion by water, wind and other natural forces are very significant for tropical countries like India leading to qualitative and quantitative degradation of the soil in land area. Thus, soil may be getting removed from agricultural land to the areas where it is not at all required, such as water reservoirs. Soil particles eroded by running water ultimately find their way into water reservoirs and such a process is called 'siltation'. Reservoirs and dams are filled with soil particles and other solid materials, because of siltation. This reduces the water storage capacity of the dams and reservoirs and thus shortens their life. Apart from the filling up the reservoirs and harbours, the suspended solids present in water bodies may block the sunlight required for the photosynthesis by the bottom vegetation. This may also smother shellfish, corals and other bottom life forms. Deposition of solid in quiescent stretches of streams impairs the normal aquatic life in the streams. Further, sludge blankets containing organic solids decompose, leading to anaerobic conditions and formation of obnoxious gases. The tremendous problem of soil erosion can be controlled by proper cultivation practices and efficient soil and forest management techniques.

The organic matter content in the sediments is generally higher than that in soils. Sediments and suspended particles exchange cations with the surrounding aquatic medium and act as repositories for trace metals such as Cu, Co, Ni, Mn, Cr and Mo. Suspended solids such as silt and coal may injure the gills of the fish and causes asphyxiation.

**4.Radioactive materials**: The radioactive water pollutants may originate from the mining and processing of ores, increasing use of radioactive isotopes, radioactive materials from nuclear reactors and nuclear power plants. The radioactive isotopes found in water include $Sr^{90}$,$I^{131}$, $Cs^{137}$, $Cs^{141}$, $Co^{60}$, $Mn^{54}$, $Fe^{55}$, $Pu^{239}$, $Ba^{140}$, $K^{40}$, $Ra^{226}$.These radioactive isotopes are toxic to life forms.

## 1.3:Heavy metal pollution, sources and its effects

The scientific world has widely accepted definition for 'heavy metals'. However, it is generally accepted that the term 'heavy' refers to metals with a specific gravity that is at least five times the specific gravity of water. The specific gravity of water is one at 4°C(39°F). Simply stated, specific gravity is a measure of the density of a given amount of a solid substance when it is compared to an equal amount of water. In general terms, a 'heavy metal' has a specific weight higher than $8gm/cm^3$.

In small quantities, certain heavy metals are nutritionally essential for a healthy life. Some of these are referred to as the trace elements (e.g., Fe, Cu, Mn and Zn). These elements, or some forms of them, are commonly found naturally in foodstuffs, fruits and vegetables, and in commercially available multivitamin products (I.O.S.H.I.C, 1999). Heavy metals are also common in industrial applications such as in the manufacture of pesticides, batteries, alloys, electroplated metal parts, textile dyes, steel, and so forth. (I.O.S.H.I.C, 1999). Many of these products are in our homes and actually add to our quality of life when properly used.

Heavy metals become toxic when they are not metabolized by the body and accumulate in the soft tissues. Heavy metals may enter the human body through food, water, air or absorption through skin when they come in contact with humans in agriculture and in manufacturing, pharmaceutical, industrial or residual settings.

**Mechanism of toxicity of metals**: To understand the toxicity of metals, the electronic configuration must be in mind. Electrons bind molecules together. They are the rivets, bolts, nails and screws of the body. Electrons are usually intended in pair as they whiz around the outside of the atoms and give stability to form of the atom or molecule. When, for any reason, these paired electrons become separated, the molecule is damaged. These damaged molecules are called "free radicals" and are highly reactive, attacking other cellular structures to grab electron in order to become paired again. Usually there are enough free electrons in the vicinity to satisfy the demands of the free radicals, but when the level of free radicals increases beyond a certain point, the cellular protective electron donating mechanism which usually keep these molecules in check is exceeded. When that happens, great numbers of these radicals are set free, all greedily looking for electrons whenever they can be found. So, when toxic metals are in body tissues, there is free radical destructive activity going on constantly resulting in rapid aging and degeneration.

The ability of metals to disrupt the function of essential biological molecules such as protein, enzyme and DNA is the major cause of their toxicity. Displacement of certain metals essential for cell by similar metal is another cause of toxicity. For example: Cadmium can substitute for the essential metal zinc in certain protein that requires zinc for their structure or function. The alteration in protein can lead to toxic consequences. In the same way, lead can substitute for calcium in the bones, and in other sites where calcium is required.(Zubair Ahmad;CPP).

**Table: 1.1**. Illustrates some of the heavy metals with their sources and hazardous effects.

| **Heavy metals** | **Sources** | **Effects** |
|---|---|---|
| Aluminium | Alum, aluminium-cooking foils, animal feed, antacids, aspirin, auto exhaust, bleached flour, cans and tins, ceramics, dental amalgams, etc. | Alzheimer's, anaemia appetiteloss, memory loss, spleen pain, stomach pain, etc. |
| Arsenic | Burning of arsenate treated building materials, coal combustion, insect sprays, | Abdominal pain, anorexia, brittle nails, chronic anemia, etc |

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| **Heavy metals** | **Sources** | **Effects** |
|---|---|---|
| Beryllium | Coal burning, manufacturing house holds products, industrial dust, etc. | Disturbance of calcium and vitamins. |
| Cadmium | Airborne industrial contaminants, batteries, ceramics, cigarette smoke, electroplating, fertilizers, welding metals, etc. | Alopoecia, anaemia, cardiovascular diseases enlarged heart, renal diseases, migraines, etc. |
| Chromium | Metal plating, cooling towers, leather tanning industries, paint industry, etc. | Severe mucosal irritation, cancer, etc. |
| Manganese | Coal mining, ceramics, dry battery cells, etc. | "Manganese psychosis" a brain disease. |
| Mercury | Adhesives, air conditioner filters, algaecides, broken thermometers, burning newspapers and building materials, soft contact lens solution, tattooing, wood preservatives, etc. | Adrenal dysfunction, allergy, alopoecia, brain damage, hearing loss, vision loss, etc. |
| Nickel | Industrial waste, kelp, margarine, nuclear device testing, tea, tobacco smoke, etc. | Haemorrhages, intestinal cancer, oral cancer, nausea, etc. |

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| **Heavy metals** | **Sources** | **Effects** |
|---|---|---|
| Lead | Battery manufacture, canned fruit and juice, cigarette smoke, electroplating, lead pipes, etc. | Abdominal pain, arthritis, blindness, Parkinson's disease, schizophrenia, unintentional weight loss. |

## 1.4: Wastewater treatment

The various methods used in sewage and industrial wastewater treatment are as follows:

1. **Preliminary treatment**. The principle objectives of preliminary treatment are the removal of gross solids such as large floating and suspended solid matter, grit, oil and grease if present in considerable quantities.

Large quantities of floating rubbish such as cans, cloth, wood and other objects present in wastewater are usually removed under preliminary treatment.

2. **Primary treatment**. After the removal of gross solids, gritty materials and excessive quantities of oil and grease, the next step is to remove the remaining suspended solids as much as possible. This is aimed at reducing the strength of the wastewater and also to facilitate secondary treatment.

The suspended matter can be removed effectively and economically by sedimentation. This process is particularly useful for treatment of wastes containing high percentage of settleable solids or when the waste is subjected to combined treatment with sewage.

Finely divided suspended solids and colloidal particles cannot be removed by simple sedimentation by gravity. In such cases, mechanical flocculation or chemical coagulation is employed. Coagulation is the most effective and economical means to remove impurities.

Sometimes, in addition to the coagulants, other chemicals called "coagulant aids" are also used in very small quantities to promote the formation of large and quick settling floc and thereby enhancing coagulation. Activated silica and polyelectrolytes such as polymers of cyanamide, acrylic acids and their derivatives, and hydrolyzed high molecular weight polymers having molecular mass $10^4$ to $10^6$ of acrylamide or acrylonitrile are the most commonly used coagulant aids.

Some industries produce different type of wastes, having different characteristics at different intervals of time. Hence, uniform treatment is not possible. In order to obviate this problem, different streams of effluents are held in big holding tanks for specified periods of time. Each unit volume of the waste is mixed thoroughly with other unit volumes of other wastes to produce a homogenous and equalized effluent.

Highly acidic and highly alkaline wastes should be properly neutralized before being discharged. Acidic wastes are usually neutralized by treatment with lime stone or lime slurry or caustic soda, depending upon the treatment and quality of the waste .Alkaline wastes may be neutralized by treatment with sulphuric acid or $CO_2$ or waste boiler flue gas.

If both acidic and alkaline wastes are produced in the same plant or at nearby plants, storing them in separate holding tanks and mutual neutralization by mixing them in appropriate proportion is the cheapest method.

**3. Secondary Treatment**. In secondary treatment, biological processes involving bacteria and other microorganisms remove the dissolved and colloidal organic matter present in wastewaters. These processes may be aerobic or anaerobic.

In aerobic processes, bacteria and other microorganisms consume organic matter as food causing coagulation and flocculation of colloidal matter, oxidation of dissolved organic matter to $CO_2$ and degradation of nitrogenous organic matter to ammonia, converted to nitrite and eventually to nitrate. Thus, secondary treatment reduces BOD. It also removes appreciable amounts of oil and phenol. However, commissioning and maintenance of secondary treatment systems are expensive.

Anaerobic treatment is mainly employed for the digestion of sludges. However, organic liquid wastes from dairy, slaughterhouses etc., were treated by this method economically and effectively. The efficiency of this process depends upon pH, temperature, waste loading, absence of oxygen and toxic materials.

**4. Tertiary Treatment**. Tertiary treatment is the final treatment, meant for "polishing" the effluent from the secondary treatment processes, to improve the quality further. The main objectives of tertiary treatment are the removal of fine suspended solids, bacteria, dissolved inorganic solids and final traces of organics.

Depending upon the required quality of the final effluent and the cost of treatment that can be afforded in a given situation, any of the following treatment methods can be employed:

**a. Evaporation**. This is an expensive process. It is used only when the recovered solids or the concentrated solutions are reused, e.g., some electroplating wastes. This method is also employed for concentrating radioactive liquid wastes.

**b. Ion exchange**. The use of ion exchange for de-mineralization of water is well known. It is widely used for obtaining deionized water for use in high-pressure boilers. This process is now extended to wastewater treatment for the removal and recovery of toxic materials from wastewater. Ion exchange process is economical only when the recovered salts are reused in the process, as in electroplating industry. Despite the simplicity of its operation, the method may not be economical if the objective of the treatment is only the removal of dissolved solids from wastewater. Special ion exchangers are available for the retrieval of toxic metal ions from industrial wastewater.

**c. Reverse osmosis**. When a wastewater containing dissolved solids is allowed to pass through a semi-permeable membrane, at a pressure over and above the osmotic pressure of the wastewater, only the water from the waste permeates through the membrane, leaving behind a concentrated liquor, containing the dissolved solids. This process is particularly suitable and effective for the removal of dissolved solids from wastewater. The cost of the membranes and the fouling of the membranes are the major limitations of this process.

**d. Chemical precipitation**. The dissolved solids in the wastewater, particularly the heavy metal ions, can be removed by precipitation as their hydroxides with cheap precipitating agent like lime. Chromates in the electroplating waste are highly toxic and can be removed by treatment with $FeSO_4$ first to reduce the chromates to Cr (III), followed by precipitation with lime.

**e. Adsorption**. Adsorption is a power tool for the environmental engineer for the removal of trace amounts of organic and inorganic contaminants present in municipal and industrial wastewater. Conventional and Non-conventional adsorbents are used in this approach. Activated carbon, a conventional adsorbent is marketed in both powdered and granular form. Activated carbon, has a great capacity for the adsorption of organic molecules, Granular activated carbon (GAC) is slightly more expensive than Powdered activated carbon (PAC) but easier to handle, easier and cheaper to regenerate. To make adsorption an economically feasible process non-conventional adsorbents have come into application, includes inorganic adsorbents, organic adsorbents and biosorbents capable of removing/retrieving of heavy metal ions from wastewater. Materials like chitin, chitosan, modified cotton, keratin, fly ash, mustard oil cake, fruit peels, barks, etc. are used as non-conventional adsorbents.

## References:

International Occupational Safety and Health Information Center; 1999

Mohammad Rafiq; Green File; Vol.1; No.5; 1995; Pagn. 55

Zubair Ahmad; Deputy Programme Coordinator (CPP); Heavy metal and mechanism of toxicity.

# CHAPTER- 2

# Survey of Literature

# 2. Survey of literature

Adsorption is one of the promising processes for the removal of organic and inorganic pollutants from water particularly if the pollutants are present in low concentrations.

A variety of adsorbents though good in adsorption efficiency but not economically feasible. In this regard, the use of non-conventional adsorbents for the removal of pollutants from the wastewater provides an upper hand over the use of conventional adsorbents.

The non-conventional adsorbents may be classified as:

**1. Inorganic Adsorbents**: They may be natural minerals, ores, clay and waste materials from various industries like fly ash, metallurgical solid wastes like bauxite and red mud
etc.

**2. Organic Adsorbents**: A large number of waste materials of organic origin like dead leaves of trees, bark, roots, seed shells and saw dust from various plants in the form of powder have been utilized for the removal of heavy metals and their adsorption properties have been explored.

**3. Biosorbents**: They include biomass of algae, fungi, and peat moss. The advantages of biosorption are low cost, high efficiency of heavy metal removal from dilute solutions, regeneration and possible metal recovery.

Various adsorbents used for the removal of organic and inorganic pollutants have been listed in the tables 2.1 and 2.2.

**Table: 2.1**. Summary of various adsorbents used for the removal of inorganic pollutants from wastewater (2000- Feb, 2005)

| Adsorbents | Metal ions removed | Remarks | References |
|---|---|---|---|
| Pyrite | Cu (II) | Oxidation is accompanied by the reduction of Cu (II) to Cu (I). | Weisener and Gerson, 2000 |
| Peat | Zn and Cd | Peat columns are able to retain the main interferent on adsorption of Zn and Cd ions in solution. | Petroni et al., 2000 |
| Fly ash | Ni ions | The removal of Ni (II) is 96%. | Ricou-Hoeffer et al., 2000 |
| Hematite | Cd (II) | 100% adsorption of Cd takes place around pH-9.5. | Gur Prasad., 2000 |
| Manganese oxide | As ions | As ion concentration was decreased to 2.3mg/l from 10mg/l in 20minutes at pH 4.5-5.0 | Kasai et al., 2000 |
| Serpentine | Cd (II),Cu (II),Fe(III) Pb (II) and Ni (II) | 99% heavy metal removal in synthetic solutions of ions | Guo et al., 2000 |
| Sawdust | Cu | Provide strong evidence to support the hypothesis of adsorption mechanism. | Yu et al., 2000 |
| *Avena monida* (Oat) *biomass* | Cr (VI) | Cr (VI) is reduced to Cr (III) in polluted water | Gardea-Torresdey et al., 2000 |
| Sawdust | Cr | 95% Cr ions were removed. | Srivastava et al., 2000 |

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|---|---|---|---|
| Surfactant modified clinoptilolite | Sulphate, Hydrogen chromate and Di hydrogen phosphate anions | The Kinetic results showed that Sulphate and Dihydrogen phosphate anions were slow processes *while Hydrogen chromate* anion was completed in few minutes. | Vujakovic et al., 2000 |
| Anaerobically digested sludge | Cd (II),Cu(II),Ni(II) and Zn (II) | Affinity of the sludge was established as Cu(II)>Cd(II)>Zn(II)>Ni(II) | Artola et al., 2000 |
| Cow dung cake | Cr (VI) | Cr (VI) removal is 90%. | Das et al., 2000 |
| Dried animal bones | Zn | - | Banat etal., 2000 |
| Red mud | As | Follows Ist Order rate expression and obeys Langmuir model .The adsorption of As (III) was exothermic and As (V) was endothermic. | Altundogan et al., 2000 |
| Iron oxide coated granular activated carbon | Cu (II) | Cu (II) adsorption capacity per gram of iron oxide on Fe-GAC adsorbents was at least three times greater than discrete Fe oxide | Fan et al., 2000 |
| Acidic Manganese chloride | Cu, Ni, Co, Pb and Fe | - | Diniz etal., 2000 |
| Tea leaves | Ni (II) and Cr (III) | Maximum adsorption were 7.97 and 5.91 mg/g. | Nishioka et al., 2000 |
| Carbon of Almond husk | Cr (VI), Cd (II) and Cu (II). | The removal of Cr (VI), Cd (II) and Cu (II) were 94.4, 93.7,94.7% respectively | Hasar and Cuci 2000 |

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|---|---|---|---|
| Effloresced coal | Pb(II),Cu(II), Zn(II) and Ni (II) | Removal rate was 97% at pH.4 and 20°C. | Mei Jianting ,2000 |
| Condensed-tannin gel | Cr (VI) | - | Nakano et al., 2000 |
| Plastic clay | Cr (VI) | Equilibrium data fitted well to Lagergreen equation . | Rai et al., 2000 |
| Lignite-based Cabon | Ni (II) and Cu (II) | Pseudo-second order process | Samra S.E., 2000 |
| Barks of eucalyptus and *Cassia fistula* | Cr and Cu | Eucalyptus bark is more efficient in removal of Cr and Cu then *Cassia fistula* | Tiwari et al., 2000 |
| Natural and modified francolite | Pb (II) | Maximum adsorption capacities were 35.625 and 1160mg/g respectively. | |
| Activated Charcoal and Coconut shell carbon | Heavy metals | Maximum removal is obtained at pH 1-3.4 at very low contact time. | Sivasamy et al., 2000 |
| Rice straw | Cr (VI) | - | Samanta et al., 2000 |
| Low cost adsorbents | Heavy metals | Tea dust was an effective replacement for activated carbon | Pari and Durai 2000 |
| Talc, Chalcopyrites and Barite | Cd ions | Follows Langmuir and Freundlich equations | Soltan , M.E.,2000 |

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|---|---|---|---|
| Polyhydroxyethyl- -methacrylate | Heavy metal ions | Maximum adsorption ratio was as high as 99% | Arpa et al., 2001 |
| Thiol cotton fibers | Heavy metals | - | Yu et al., 2001 |
| Bone char | Cd, Cu and Zn ions | Sorptions of Cd and Zn ions onto bone char are primarily film-pore diffusion controlled. | Cheung et al., 2001 |
| Activated carbon | Metal ions | Langmuir isotherm fit the data better in single component systems. | Mohan and Chander 2001 |
| Activated carbon from fertilizer waste | Hg (II) | Hg(II) adsorption increased with a decrease in pH; the process is exhothermic. | Mohan et al., 2001 |
| Nitrified lignite | Cr | The adsorption isotherms were consistent with Langmuir equation. | Wang et al., 2001 |
| Red mud | Pb and Cr | Thermodynamic parameter indicates the fessibility of the process. | Gupta et al., 2001 |
| Aluminium based coagulant | As | Solution As (V) is converted to particulate As predominantly by clarification. | Gregor, J., 2001 |
| Modified Corncobs | Metal ions | - | Vaughan et al., 2001 |

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|---|---|---|---|
| Polyurethane foam sorbent coupled with Ammonium pyrrolidine dithioca-rbamate(APDC) | Hg, Cd and Pb | - | Anjaneyulu et al., 2001 |
| Chitosan impregnated with microemulsion. | Cr | Uptake process obeys the Langmuir isotherm. | de Dantas et al., 2001 |
| Poly (ethylene imine) immobilized Poly (methyl methacrylate) microspheres. | Cu, Cd, and Pb | Immobilization of adsorbent significantly increased the heavy metal adsorption (0.224 mmol/g for Cu (II),0.276 mmol/g for Cd(II),and 0.126mmol/g Pb(II). | Duru et al., 2001 |
| Chitosan | Cr (VI) | - | Tang et al., 2001 |
| Wollastonite | Cr (VI) | Maximum adsorption (74.4%) was at pH 2.0 temperature 30°C and minimum (12.3%) at pH 8.0. | Sharma, Y.C., 2001 |
| Modified saw dust | Cr (VI) | Maximum removal (>99%) was obtained at initial concentration 25 mg/l at pH 2-3. | Unnithan and Anirudhan .,2001 |
| Saw dust | Heavy metals | - | Yu et al., 2001 |
| Nätural zeolite | Heavy metals | Basic adsorption models except the Loading ratio correlation model could describe multi--species ion exchange equilibrium for heavy metals/natural zeolite systems well. | Lee and Moon. , 2001 |

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|---|---|---|---|
| *Aspergillus niger* biomass | Pb (II) | Follows first order reaction kinetics. | Jianlong et al., 2001 |
| Cauliflower leaves | Cr (VI) | At room temperature leaves show maximum adsorption properties.As the pH of effluent increases the adsorption properties decreases. | Farooqui and Kotharkar, 2001 |
| Fly ash | Cu | Maximum percentage of adsorption is obtained at a pH-5. | Hajarnavis and Bhide, 2001 |
| Sandstone | Pb (II) and Zn (II) | Adsorption capacity of sandstone to Pb (II) was similar to that of Breccia,But the adsorption capacity of Breccia to Zn (II) was greater than that sandstone. | Xu et al., 2001 |
| *Durvillaea potatorum* ,a marine alga. | Cu (II) | | Yu and Kaewsarn, 2001 |
| Synthetic Zeolites | Pb (II) | | Karetina et al., 2001 |
| Activated Bentonite | Pb (II) | Obeyed Langmuir isotherm behavior,and ion exchange, surface complex were the main adsorption forms. | Xia and Shi, 2001 |
| Soil collected from sewage sludge | Cd | | Pandeya et al., 2001 |
| Chitosan / PVA Hydrogel Beads | Pb | | Jin and Bai., 2002 |

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|---|---|---|---|
| Poly (hydroxyethyl methacrylate) adsorbents with Thiazolidine Groups | Hg (II) | The maximum desorption ratio was as high as 99%. | Arpa et al., 2002 |
| [0.5M $SiO_2$: 0.5M Fe $(OH)_3$] | Metal ions | | Mustafa et al., 2002 |
| Chicken Feathers | Cu, Zn and Ni ions | | Al-Asheh et al., 2002 |
| Solar thermal and Chemo thermal Activated Carbon | Pb (II) and Cr (VI) | | Nagar and Singh, 2002 |
| *Garcinia cambogia*,a Plant Biomass | Cr | | Chanderasekhar et al., 2002 |
| *Rhizopus oligosporus* | Pb (II) | | Xia and Liyun, 2002 |
| Rice Husk Carbon | As (III) | | Nagarnaik et al, 2002 |
| Dithiocarbamate Grafted on Silica Gel | Complexed Mercury | | Venkatesan et al., 2002 |
| Activated Carbon | Cr (III) | | Milich et al., 2002 |
| Pillared Clays and Iron oxides | As | | Lenoble et al., 2002 |
| $TiO_2$ loaded Amberlite XAD-7 Resin | As (III) and As (V) | | Balaji and Matsunaga, 2002 |
| Peat | Cd | | Ulis et al., 2002 |
| Methacrylamidocysteine containing Porous Poly (hydroxyethylmethacrylate) Chelating Beads | Heavy metal ions | Maximum adsorption capacity is observed for 1058.2 mg/g for Cd (II). | Denizli et al., 2002 |
| Porous Chelating Resin Containing Polymer Ligand | Metal ions | | Tanco et al., 2002 |

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| Crosslinked chitosan | Hg and Precious metals | | Oshita et al., 2002 |
| Brucite | Metal ion sorption | | Izotov et al., 2002 |
| Fly ash, Slag, Ordinary Portland Cement and Related Blends | Phosphate ions | | Agyei et al., 2002 |
| Hybrid Macroporous Materials | Heavy metal ions | | Schroden et al., 2002 |
| Natural Zeolites | Radioactive Iodine | | Faghihian et al., 2002 |
| Activated Carbon | Au-CN species | | Mc Grath et al., 2002 |
| Geothite-coated sand | Cd | | Lai et al., 2002 |
| Natrolite and Clinoptilolite Rich Tuffs | Cr (III) | | Dahbi et al., 2002 |
| Natural Sorbent | Cr(III) and Fe(III) | | Farias et al., 2002 |
| Bone Charcoal | Mo ions | | Faghihian et al., 2002 |
| Silicon Hybrid Surface | Toxic Metals | | Meunier et al., 2002 |
| Acid-pretreated *Chlorella vulgaris* | Cu (II) and Ni (II) | | Mehta et al., 2002 |
| Iron Coated Sand | As | | Petrusevski et al, 2002 |
| Fly Ash of Poultry Litter | Cr (III) | | Kelleher et al., 2002 |
| Olivine Process Dust | Cu | | Kleiv and Sandvik , 2002 |

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| Turkish Fly Ashes | Ni(II),Cu(II), Zn(II) | | Belgin Bayat, 2002 |
| Turkish Fly Ashes | Cr(VI) and Cd(II) | | Belgin Bayat, 2002 |
| Amino-Functionlized MCM-41 and SBA-1 | Chromate and Arsenate | | Yoshitake et al., 2002 |
| Kaolinite | Pb and Cd | | Coles and Yong, 2002 |
| Iron Oxide and Kaolin | Cu (II) and Ni (II) | | Sen etal., 2002 |
| Paper Mill Sludges | Heavy Metals | | Calace et al., 2002 |
| Sawdust | Heavy Metals | | Shukla et al., 2002 |
| Fe, Mn Oxides and Humic Acid | Au (I, III) Complexes | | Ran et al., 2002 |
| WD-ekstra Activated Carbon | Cu and Pb ions | | Kapica et al., 2002 |
| Hydrous Ferric Oxides | Transition metal ions | | Khalil et al., 2002 |
| BoneCharcoal | Cr (III) | 90% Cr removal | Dahbi et al., 2002 |
| *Volvariella volvacea* | Cu and Zn | | Mohanty and Chaudhury, 2002 |
| Ion Exchange Resinsfrom Ammonical Thiosulfate | Au and Cu | | Zhang and Dreisinger2002 |
| Activated Carbon | Sb and Ar | | Navarro and Alguacil. , 2002 |
| Chitosan Derivatives | Cu (II) | | Baba et al., 2002 |
| ACCs | Cu (II), Ni (II) and Pb (II) | | Faur-Brasquet et al., 2002 |

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|---|---|---|---|
| *Aspergillus niger* and *Penicillium austurianum* | Cd | | Rostami and Joodaki. 2002 |
| Low-cost Adsorbents | Cr (VI) | | Dakiky et al., 2002 |
| Activated Carbon Cloths | Metal ions | | Faur-Brasquet et al., 2002 |
| Perlite | Cd | | Mathialagan and Viraraghavan, 2002 |
| Granular Activated Carbon | Zn and Cd ions | | Choi and Kim., 2002 |
| Granulated Iron Hydroxide (GEH) | As | | Wingrich and Wolf., 2002 |
| Sludge Ash | Ni (II) | | Chih-Huang Weng, 2002 |
| Hydrous Al $(OH)_3$ in presence of a range of Chelates | Cd(II) | | Burnett et al., 2002 |
| MgO(100) | Metal ions | | Campbell and Starr., 2002 |
| Benzophenone | Cu | | Lee and Choi., 2002 |
| Poly (N-vinyl formamide/ Acrylonitrile)Chelating Fibers | Heavy Metal Ions | | Lin et al., 2002 |
| Keratin Composed Biosorbents | Heavy metals | Freundlich isotherm model was found to be applicable. | Banat et al., 2002 |

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| Activated and Non-activated Date-pits | Cu and Zn ions | Adsorption capacities for the non-activated date-pits towards $Cu^{2+}$ and $Zn^{2+}$ ions as high as 0.15 mmol/g and 0.09 mmol/g, respectively, | Banat et al., 2002 |
| Activated Carbons | Mercury (II) Ions | | Kannan and Rajakumar, 2003 |
| Aquatic Plant (Myriophyllum spicatum) | Heavy Metal | | Keskinkan et al., 2003 |
| Modified Activated Carbons | Transition metal ions (Cu, Zn, Ni and Cd) | | Saha et al., 2003 |
| Freshwater alga *Chlorella kesslerii* | Pb | | Slaveykova and Wilkinson., 2003 |
| Olive Mill Residues | Copper | | Veglio et al.,2003 |
| Orange Waste | Arsenate and Arsenite Anions | | Ghimire et al., 2003 |
| Chitosan | Mercury Ion | | Jeon and Holl., 2003 |
| Clay soils | Lead, copper and zinc | | W. Y. Wan Zuhairi .,2003 |
| Bentonite | Ni (II) | | Tahir and Rauf., 2003 |
| Ferrous Saponite | Cr(VI) | | Parthasarathy et al., 2003 |
| Humic Acids Extracted from Brown Coals | Metal Ions | | Martyniuk and Wickowska.,2003 |
| Activated Carbon from Almond Husks | Zinc (II) | 92% removal of Zn (II) was achieved. | Hasar et al., 2003 |

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| Natural and Modified Radiata Bark Pine | Copper | | Montes et al., 2003 |
| Cellulose Graft Copolymers | Heavy Metal Ions | | Guclu et al., 2003 |
| Savanna Acid Soil | Copper | More than 65% of applied Cu was sorbed at pH ≥ 3.0, far below the point of zero net charge of the soil. | J. O. Agbenin. 2003 |
| Recycled-wool-based Non woven Material | Lead Cations | | Radetic et al., 2003 |
| $SO_2$Treated Activated Carbon | Cadmium | | Macias-Garcia et al., 2003 |
| Olive Pomace | Heavy Metal | | Pagnanelli et al., 2003 |
| Chitosan | Metal Ions | | Navarro et al., 2003 |
| Concrete Particles | Silver | | Shakila Begum., 2003 |
| Hydrophobized Zeolitic Media | Arsenate and Chromate | | Eva Chmielewska 2003 |
| Thai Kaolin and Ballclay | Heavy Metal | Adsorption of metals in the mixture solutions by kaolin was: Cr > Zn > Cu ≈ Cd ≈ Ni > Pb, and for ballclay was: Cr > Zn > Cu > Cd ≈ Pb > Ni. | Chantawong and Harvey., 2003 |
| Natural and Modified Zeolitic Minerals | Mercury Ions | Retention of Hg was highest for zeolitic minerals treated with organic compounds. | Gebremedhin-Haile and Olguin. 2003 |

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| Zeolites synthesized from Fly ash | Heavy Metals | The adsorption capacity of zeolites synthesized is higher than that of fly ash. | Yanxin et al., 2003 |
| Fungus *Penicillium canescens* | Heavy Metal Ions | The maximum adsorption capacities of the heavy metal ions studied onto the fungal biomass under non-competitive conditions were 26.4 mg/g for As (III), 54.8 mg/g for Hg (II), 102.7 mg/g for Cd (II) and 213.2 mg/g for Pb (II), respectively. | Say et al., 2003 |
| Synthetic Zeolites | Zn (II) | | Badillo-Almaraz et al., 2003 |
| Bagasse Fly Ash | Cadmium and Nickel | | Gupta et al., 2003 |
| Natural Condensed Tannin | Lead | | Zhan and Zhao., 2003 |
| Sea Nodule | Lead | | Bhattacharjee et al., 2003 |
| Chitosan | Lead | | Ng et al., 2003 |
| Chemically-treated Chicken Feathers | Copper and Zinc Ions | | Al-Asheh and Banat , 2003 |
| Grafted Silica | Cu (II) and Pb (II) | | Chiron et al.,2003 |
| Calcined Mg-Al-$CO_3$ Hydrotalcite | Chromium (VI) | | Lazaridis and Asouhidou , 2003 |
| Neutralized Red Mud | Arsenate | | Genc et al., 2003 |
| Low-cost Adsorbents | Heavy Metal Ions | | Wang et al., 2003 |

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| Sheep Manure | Copper and Cadmium Ions | . Maximum uptakes for 100 ppm $Cu^2$ and 100 ppm $Cd^2$ ions were found to be 17.8 mg g and 10.8 mg g, respectively. The equilibrium uptakes for both copper and cadmium ions were attained within the first 10 min. | Kandah et al., 2003 |
| Organosolv Lignin | Copper (II) | | Acemiolu et al., 2003 |
| Sepiolite | Cu (II) and Zn (II) | | L. I. Vico , 2003 |
| Goethite | Phosphate and Arsenate | | Gao and Mucci, 2003 |
| *Auricularia polytricha* | Copper | Bio sorption capacity of about 90% was obtained. | Galli et al., 2003 |
| Functionalized Silica | Heavy Metal Ions | | Bois etal., 2003 |
| Kaolinites | Arsenic (V) | | Cornu et al., 2003 |
| Rice bran | $Ca^{2+}$ | The value of $pH_0$ , at which the surface charge is zero was found to be 5.5. | Navas and Carrasquero-Duran, 2003 |
| Maple Sawdust | Chromium | | Yu et al., 2003 |
| Anilinepropylsilica Xerogel | Cu (II) | | Pavan et al., 2003 |
| Activated Carbon from Furfural | Mercury (II) | | Yardim et al., 2003 |
| Iron Oxyhydroxide | Hexavalent Uranium | | Wazne et al.,2003 |
| Sewage Sludge Ash | Copper | | Pan et al., 2003 |

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| Clinoptilolite | $Cu^{2+}$, $Fe^{3+}$ and $Cr^{3}$ | | Inglezakis et al., 2003 |
| Silica Gel | Cesium | | Bascetin et al., 2003 |
| Amino Acids Modified Chitosans | Copper Ion | | Dehonor-Gomez et al.,2003 |
| Goethite | Copper, Nickel, and Cadmium | | Buerge-Weirich and Behra , 2003 |
| Sol-Gel Silica Doped with 1-(2-Pyridylazo)-2-Naphthol | Cd Ions | It was observed that a sol gel loaded with 0.09 mmol PAN/g, had a capacity of 0.044 mmol Cd/g | Khan et al., 2003 |
| *Penicillium chysogenum* mycelium | $Ni^{2+}$ | The adsorption capacity for $Ni^{2+}$ on to the surface molecular imprinting adsorbent on *Penicillium chysogenum* mycelium (the surface-imprinted adsorbent) was 40-45 mg $g^{-1}$ (using 200 mg $Ni^{2+}$ $l^{-1}$), two times of the mycelium adsorbent. | Su and Wang, 2003 |
| Mineral Matrix of Tropical Soils | Heavy Metals | Ultisol and Alfisol soils showed the highest maximum adsorption values, in the order of 50.76 and 64.52 mmol $kg^{-1}$, whereas some Oxisols showed the lowest values, in the order of 23.92 and 30.86 mmol $kg^{-1}$. | Fontes and Gomes, 2003 |
| Kaolinite and Kaolinite–humic Acid Complexes | Arsenic (V) | | Saada et al., 2003 |

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| Ce(IV)-doped Iron Oxide | Arsenic (V) | | Zhang et al., 2003 |
| Gibbsite | As (III) | | Weerasooriya et al., 2003 |
| Montmorillonite-Al hydroxide | Zinc ion | | Janssen et al., 2003 |
| Rare earth metal-doped iron oxide | Arsenic (V) | The cerium (Ce(IV))-doped adsorbent (CFA4) has the highest adsorption capacity. | Yu et al., 2003 |
| Alumina or Chitosan | Heavy metals | | Cervera et al., 2003 |
| Low-Rank Coal (leonardite) | Zn | Effective removal of $Zn^{2+}$ was demonstrated at pH values of 5-6. | Sole and Casas, 2003 |
| Maize cell walls | Boron | Adsorption increased with increasing solution pH from 4.5 to 10, exhibited an adsorption maximum at pH 10-10.5, and decreased with increases in pH above 10.5. | Sabine Goldberg 2003 |
| Goethite | Mercury and Cadmium | | Backstrom et al., 2003 |
| Indigenous Low-cost Material | Cr (VI) | | Y. C. Sharma, 2003 |
| Gellan Gum Gel Beads | Heavy Metal | | Lazaro et al, 2003 |
| Hydrolyzed Polyacrylonitrile Fibers | Copper | | Deng et al., 2003 |
| Goethite | Uranium (VI) | | Missana et al., 2003 |

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| Thiol Functional Adsorbent | Mercury (II) | | Nam et al., 2003 |
| *Pseudomonas Putida* | cadmium, copper, lead and zinc | 80% removal for all metals studied.The process of biosorption can be described by a Langmuir-type adsorption model | Pardo et al., 2003 |
| Modifications of Furfural | Mercury | | Budinova et al., 2003 |
| Kaolinite, Illite and Montmorillonite | Barium | | Atun and Bascetin 2003 |
| Dithiocarbamate grafted on mesoporous silica | Complexed mercury | The enthalpy change accompanied by the sorption of mercury was found to decrease from 83.7 to 6.2 kJ/mol, when the initial concentration of mercury was increased from 5 $10^{-4}$M to 1.5 $10^{-3}$M. | Venkatesan and Srinivasan, 2003 |
| Chitosan | Copper (II) | | Cheung et al., 2003 |
| Rhizopus arrhizus | Cr (VI), Cu(II), and Cd(II) Ions | | Sa et al., 2003 |
| Calcined Zn/Al Hydrotalcite-like Compound (HTlc) | Fluoride | | Das et al., 2003 |
| Loess with high carbonate content | Copper | | Ni Jinren, 2003 |
| Palm Fruit Bunch and Maize Cob | Iron and Manganese | The adsorbents used removed the studied metal ions effectively. | Nassar et al., 2003 |

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| Activated and Non-activated Oak Shells | Copper | The $Cu^{2+}$ ion uptake by oak shells increased with decreasing sorbent concentration or with an increase in $Cu^{2+}$ ion concentration or solution pH. | Al-Asheh et al., 2003 |
| Novel Dye-doped Sol-Gel Silica | Cobalt | The maximum adsorption of Co (II) ions onto the TAR-doped sol-gel silica from single solutions was 12.6 μmol/g. | Khan et al., 2003 |
| Date Pits | Cadmium Ion | The kinetic data for the adsorption process obeyed a second-order rate equation. | Banat et al., 2003 |
| Micaceous Mineral of Kenyan Origin | Cu (II) | An adsorption capacity of 0.850 g/g was achieved for MicaM towards the $Cu^{2+}$ ion. | Attahiru et al., 2003 |
| Powdered Marble Wastes | Copper (II) | 100% $Cu^{2+}$ ions was attained | Ghazy et al., 2003 |
| Silica Gel | $Zn^{2+}$, $Ni^{2+}$ and $Cd^{2+}$ ions | The selectivity of the solid was observed to be in the order $Zn^{2+} > Ni^{2+} > Cd^{2+}$. | Mustafa et al., 2003 |
| Clinoptilolite Mineral | Lead, Barium | | Cakicioglu-Ozkan and Ulku, 2003 |
| Surfactant-modified Zeolites | Radioactive Iodide | Modified forms exhibited an adsorption capacity much higher than those of the respective natural samples. | Faghihian et al., 2003 |
| Sugarcane Bagasse Pith | Cadmium (II) | | Krishnan and Anirudhan, 2003 |

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| Granular Ferric Hydroxide | Arsenic | | Thirunavukkarasu et al .,2003 |
| Vineyard soils of Geneva | Copper | | Celardin et al., 2003 |
| Peat | Metal Ions | Good agreement between the predicted theoretical breakthrough curves and the experimental results is observed. | Ko et al., 2003 |
| Chicken Feathers | Heavy Metals | | Al-Asheh et al., 2003 |
| Galena (PbS) and Sphalerite (ZnS) | Arsenite | Arsenite sorbed appreciably only at pH > ~5 for PbS and pH ~4.5 for ZnS, behavior distinct from its adsorption on other substrates. | Bostick et al., 2003 |
| Na-montmorillonite | Heavy Metals | | Abollino et al., 2003 |
| Natural Materials | Lead Ions | | Abdel-Halim et al., 2003 |
| Calcium Alginate Beads Containing Humic Acid | Chromium | | Pandey et al., 2003 |
| Alunite | Phosphate | | Mahmut Ozacar, 2003 |
| High-performance Activated Carbons | Chromium | | Hu et al., 2003 |
| Activated Carbon by Complexation with Surface Functional Groups | Lead (II) and Copper (II) | | Pesavento et al., 2003 |

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| Vegetated Filter Strips | Phosphorus | The average phosphorus trapping efficiency of all vegetated filters was 61% and ranged from 31% in a 2-m filter to 89% in a 15-m filter. | Abu-Zreig et al., 2003 |
| Rice Straw | Selenate | | Zhang and Frankenberger Jr, 2003 |
| 2-aminothiazole-modified silica gel | Cu, Ni, and Zn | The adsorption capacities of SiAT determined for each metal ion were (mmol $g^{-1}$): Cu (II) =120, Ni (II)=110 and Zn (II)=090 | Roldan et al., 2003 |
| Organic Manure | Copper | | Bolan et al., 2003 |
| *Penicillium chrysogenum* | Metal Ions | | Tan and Cheng, 2003 |
| A 1.10 Phenanthroline-grafted Brazilian Bentonite | Cu Ions | | De Leon et al., 2003 |
| Caustic Treated Waste Baker's Yeast Biomass | Copper Ions | | Goksungur et al., 2003 |
| Amberlite IR-118H resin | Uranium | | Kilislioglu and Bilgin, 2003 |
| Activated Carbon | Pb (II), Cd (II), and Cr (VI) | | Rivera-Utrilla et al., 2003 |
| Pseudomonas Putida 5-x Isolated from Electroplating Effluent | $Cu^{2+}$ | | Wang et al., 2003 |
| Turbid River water | Cu and Ni | | Herzl et al., 2003 |

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| Regenerated Sludge from a Water Treatment Plant | Copper and Lead Ions | | Wu et al., 2003 |
| Chitosan | $Zn^{2+}$ | | Zhiguang et al., 2003 |
| Sand | Heavy Metals | | Awan et al., 2003 |
| Tea Leaves and Coffee Beans | Mercury | | Kiyohara et al., 2003 |
| Low Cost Materials | Iron and Manganese | | Nassar et al., 2003 |
| Iron-Conditioned Zeolite | Arsenic | | Onyango et al., 2003 |
| Carbonaceous Materials Prepared from Bamboo and Coconut Shell | Nitrate Anion | | Ohe et al., 2003 |
| Sawdust Carbon | Arsenic (III) | | Nagarnaik et al., 2003 |
| Vaterite Modification of Calcium Carbonate | $^{90}Sr$ and $^{90}Y$ | | Berdonosov and Berdonosova,2003 |
| Activated Coke from Wood Pellet | Hydrogen Sulfide | | Mitomo et al., 2003 |
| Iron-Modified Zeolite | Arsenic | | Onyango et al., 2003 |
| Sepiolite | Cobalt | | Kara et al., 2003 |
| Activated Carbon from Acidic Media: Nitrate and Sulfate Media | Silver | | Jia et al., 2003 |

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| Iron Oxide-Coated Sand | Arsenic | The batch and column studies showed that iron oxide-coated sand filtration could be effectively used to achieve less than 5 μg $L^{-1}$ As in drinking water. | Thirunavukkarasu and Viraraghavan 2003 |
| Low Cost and Waste Material | Copper and Cadmium Ions | Of all the adsorbents studied, bentonite and compost presented the highest removal efficiencies, reaching 99% for copper when cadmium is also present, for initial solution concentrations of up to 100 mg $L^{-1}$. | Ulmanu et al., 2003 |
| Bone Charcoal | Cu and Zn | | Wilson and Pulford, 2003 |
| Activated Carbon from Coconut Coirpith | Cd (II) | | Kadirvelu and Namasivayam,2003 |
| Sulfate-modified Iron Oxide-coated Sand (SMIOCS) | Arsenic (III) | | Vaishya and Gupta, 2003 |
| Muloorina Illite and Related Clay Minerals | Cd(II) | | Lackovic et al., 2003 |
| Crosslinked chitosan | Vanadium (V) and Tungsten (VI) | | Qian et al, 2004 |
| Activated carbon | Sulfur and nitrogen | | Sano et al, 2004 |
| Calabrian Pine Bark | Fe(II) | | Bilal Acemiolu, 2004 |

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| *Lactuca Sativa* L. cv. *Ostinata* | Cd (II) | | Waisberg et al, 2004 |
| Sandy Loam Soil | Pb (II) | | Chih-Huang Weng, 2004 |
| Silica-dithizone | Hg (II) | | Cestari et al, 2004 |
| Activated Neutralised Red Mud | Arsenic | | Genc-Fuhrman et al, 2004 |
| *Chryseomonas luteola* TEM05 | Cr and Al | | Ozdemir and Baysal, 2004 |
| Natural Zeolite | Zn,Cu and Pb | | Peri et al, 2004 |
| Aluminium oxide | Ferrocyanide | | Bushey and Dzombak, 2004 |
| Cross linked Alginate Gel Beads | Cu and Mn | | Gotoh et al, 2004 |
| Alginate-Chitosan Hybrid Gel Beads | Divalent Metal ions | | Gotoh et al, 2004 |
| Geothite | Heavy Metal Cations | | Kosmulski and Mczka, 2004 |
| *Ceratophyllum demersum* | Heavy Metal | | Keskinkan et al, 2004 |
| *Ecklonia maxima* | Heavy Metal | | Feng and Aldrich, 2004 |
| *Streptomyces coelicolor* A3(2) | Ni(II) and Cu(II) | | Ozturk et al, 2004 |
| Tree Fern | Cd(II) | | Ho and Wang,2004 |
| Brown,Green and Red Seaweeds | Cd | | Hashim and Chu, 2004 |
| Wood Saw dust | Heavy metal ions | | Sciban and Klasnja, 2004 |

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| Chinese Reed (*Miscanthus sinensis*) | Cr (III) | | Namasiyam and Holl, 2004 |
| Activated alumina | Flouride | | Ghorai and Pant, 2004 |
| *Chlorella vulgaris* | Cu | | Chu and Hashim, 2004 |
| Bone char | Metal ions | | Choy et al, 2004 |
| Hematite | Phosphate | | Xiao Huang, 2004 |
| Neutralized Red Mud (Bauxsol) | Arsenate | | Genc-Fuhrman et al, 2004 |
| Bagasse Fly Ash | Pb and Cr | | Gupta and Ali,2004 |
| Savanna Alfisol | Cu and Zn | | Agbenin and Olojo, 2004 |
| Sepiolite | Ammonium ion | | Suna Balci, 2004 |
| Iron oxide Tailings | Phosphate | | Zeng et al, 2004 |
| Treated Sawdust | Cr | | Garg etal, 2004 |
| Zn(IV) substituted ZnAl/MgAl-layered Double Hydroxide | Cr(VI) and Se(II) | | Das et al, 2004 |
| Humic substance | Cu(II) | | Alvarez-Puebla et al, 2004 |
| Kaolinite and Muloorina Illite | Cd(II) | | Lackovic et al, 2004 |
| Aluminium Impregnated Mesoporous Silicates | Phosphate | | Shin et al, 2004 |
| Montmorillonite | Quaternary Ammonium salts | | Kozak and Domka, 2004 |

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| Activated Rice Husk and Activated Alumina | Cr (VI) | | Bishnoi et al, 2004 |
| Hazelnut shell | Cr(VI) | | M.Kobya, 2004 |
| Alumina particles | Ni(II) | | Hong etal, 2004 |
| Surfactant Modified Zeolite | Chromate | | Zhaohui Li, 2004 |
| *Fontinalis antipyretica* | Cd(II) and Zn(II) | | Martin et al, 2004 |
| Fe-modified Steam Exploded Wheat Straw | Cr (VI) | | Chun et al, 2004 |
| Alginate coated Loofa Sponge Disc | Cd | | M.Iqbal, 2004 |
| Peat | Cu | | Petroni and Pires, 2004 |
| Poly acrylonitrile-immobilized dead cells of *Saccharomyces Cerevisiae* | Cu(II) | | Godjevargova and Mihova, 2004 |
| Montmorillonites | Cu | | Ding andFrost, 2004 |
| Herbaceous Peat | Cu (II) | | Gundoan et al,2004 |
| Mangenese Dioxide Complex | Co, Ni, Cu, and Zn | | Kanungo et al, 2004 |
| γ- type Alumina Particles | Ni (II) | | Hong et al, 2004 |
| Bone char | Metal ions | | Ko et al, 2004 |
| Phosphoric Acid Activated Carbon | Heavy metal ions | | Puziy et al, 2004 |

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| Granular Activated Carbon | Fe(III)andFe(III)NTA Complex | | D.S.Kim, 2004 |
| Activated Carbon | Hg | | Ho et al, 2004 |
| Geothite | Cadmium and Phosphate | | Wang and Xing, 2004 |
| Chitosan | Cu | | Wan et al, 2004 |
| Aquatic Moss *Fontinalis antipyretica* | Zn(II) and Cd(II) | | Ramiro et al, 2004 |
| Moroccan Stevensite | Metal ions | | Benhammon et al, 2005 |
| Extracellular polysaccharides(EPS) Produced by activated sludge bacterium *Chryseomonas luteola* TEM05 | Cu(II) and Ni(II) | The maximum adsorption capacity in Langmuir isotherm for calcium alginate ,calcium alginate+EPS, calcium alginate+*C.luteola* TEM05, calcium alginate+*C.luteola* TEM05+EPS were 1.505,1.989,1.976,1.937 mmol/g dry weight of Cu(II) and 0.996,1.224,1.078,1.219 mol/g dry weight of Ni(II),respectively.pH . 6.0, temperature 25°C | Ozdemir and Manav, 2005 |
| Low cost biological wastes and vermiculite | Cr | | Sumathi et al., 2005 |
| Moroccan Stevensite | Mercury (II) and Chromium (VI) | | Benhammou et al., 2005 |
| Amberlite IR-120 Synthetic resin | Cu, Zn, Ni, Pb and Cd ions | | Demirbas et al., 2005 |

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| Chitosan-based Polymeric Surfactants | Hexavalent Chromium | | Lee et al., 2005 |
| Activated Charcoal | Erbium Ions | | Riaz Qadeer, 2005 |
| Coffee Grounds as Vegetable Biomass | Lead Ions | | Tokimoto et al., 2005 |
| Sea Nodule Residues | Lead | | Agrawal et al.,2005 |
| Activated Carbons from Organic Sewage Sludge | Mercury | | Zhang et al., 2005 |
| *Thuja Oriantalis* | Cr(VI) | | Ensar Oguz, 2005 |
| Montmorillonite | Zn(II) | | Ikhsan et al, 2005 |
| Mulch | Heavy Metals | | Jang et al, 2005 |
| Etanol treated Baker's Yeast Biomass | Cadmium and Lead Ions | The maximum metal uptake values ($q_{max}$ ,mg/g) were found as 31.75 and 60.24 for Cd(II) and Pb(II),respectively.The competitive biosorption capacities of the yeast biomass for all metal ions were found to be lower than in non competitive conditions | Goksungur et al., 2005 |
| Black gram husk (BGH) | Pb,Cd,Cu,Ni,and Zn | The maximum amount of heavy metals($q_{max}$) adsorbed at equilibrium was 49.97,39.99,33.81,25.73 and 19.56 mg/g BGH biomass for Pb Cd,Zn,Cu and Ni, respectively | Saeed et al, 2005 |

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|---|---|---|---|
| Alluvial Gravel Aquifer Media | Bacteria-facilitated Cd transport | Bacteria-facilitated transport of heavy metals may pose threat to ground water quality in sites such as land fills and following disposal of industrial and domestic effluent and sludge | Pang etal, 2005 |

**Table2.2:** Summary of various adsorbents used for the removal of organic pollutants from wastewater (2000-Feb, 2005)

| Adsorbent | Organic pollutant | Remark | Reference |
|---|---|---|---|
| Coated Activated Carbon | Nitrobenzene, Benzoic acid, Phenol and Aniline | | Koh and Nakajima., 2000 |
| Rice bran | Colour based effluents. | First order reversible kinetics. The process is spontaneous in nature. | Kesarwani et al., 2000 |
| Fly ash | Dyeing waste water | Decolourization efficiency reached 91-99% under conditions of fly ash dosage 0.04-0.08 g/ml, adsorption time 3 hrs ,pH –2-10 and dye concentration 10-600mg/l. | Yan et al., 2000 |
| Carbon from Palm seed coat | o -Cresol | 95% removal of o-Cresol. Freundlich adsorption isotherm gives calculated adsorption capacity of 19.58 mg/g. | Rengaraj et al., 2000 |
| Bagasse fly ash | Rhodamine B and methylene blue | Thermodynamic parameter indicated feasibility of the process. | Gupta et al., 2000 |
| Long flame coal of Donbas and Carbon Sorbents | Petroleum products | | Stupin et al., 2000 |
| *Aspergillus niger* | Acidic and Basic dyes | Follows first order kinetic expression and the Langmuir equation. | Anjaneyulu and Bindu., 2000 |

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|---|---|---|---|
| Bagasse fly ash | 2,2-Bis (4chlorophenyl)-1,1-dichloroethane and2,2-Bis (4-chlorophenyl)-1,1-dichloroethene | < 93% removal at pH-7. | Gupta and Ali, 2001 |
| Activated carbon from coconut shell | Methylene blue | The 150µm particle size gave better adsorption than 355µm particle size | Gimba et al., 2001 |
| Recycled Alum Sludge (RAS) | Textile dye waste | RAS is good way of Removing hydrophobic dye from wastewater. RAS is not recommended for removing hydrophilic dye. | W.Chu, 2001 |
| Kaolinite | Sodium oleate | The adsorption process on Kaolinite is found to follow a two step-first order kinetic rate equation with two different($K_1$ and $K_2$ ) rates constants. | Xu et al., 2001 |
| Granular Activated Carbon and Natural Zeolites | Basic dyes | | Meshko et al., 2001 |
| Orange peel | Acid Violet 17 | The adsorption capacity was 19.88mg/gm at initial PH 6.3 | Shivaraj et al., 2001 |
| Natural porous coal | Aromatic compounds | | Tarasevich, Y.I., 2001 |

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|---|---|---|---|
| Saw dust | p-nitrophenol | Intraparticle diffusion had a hindering effect on the adsorption | Dutta et al., 2001 |
| Rice Bran | Organochlorine compound and Benzene | | Atsuko et al., 2001 |
| Organo-minerals | Phenols, benzene and toluene | | Koh and Doxon 2001 |
| Activated carbons | Cationic and Anionic dyes | | Al-Degs et al., 2001 |
| Rice husk carbon | Basic dyes | Removal of dyes was found to increase with increase in dye concentration and pH of the solution. | Singh and Srivastava, 2001 |
| Penecillium species GX2 | Anthraquinone dyes | GX2 has high adsorption capacity even at high dye concentration. | Xin et al., 2001 |
| Japanese cedar bark | Trichloroethylene | The adsorption capacity of the bark pyrolyzed at 900°C was 5 times larger than that of common activated carbon | Kurimoto et al., 2001 |
| Coir pith | Dyes | Follow first order kinetics. | Namasivayam et al., 2001 |
| Anoin-exchange resins | Carboxylic acids | | Kanazawa et al., 2001 |
| Muscovite and Hematite | Natural organic matter | | Namjesnik and Maurice, 2001 |
| Activated Carbon Fiber | Dye | | Ko et al., 2002 |

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|---|---|---|---|
| natural and synthetic adsorbents | limonin and naringin from orange juice | The estimated values of free energy of adsorption, lower than -133 kJ $mol^{-1}$ $K^{-1}$, indicate that a physiosorption process occurred | Ribeiro et al., 2002 |
| Activated Bleaching Earth | Paraquat | | Tsai et al., 2002 |
| Organic Compounds | Activated Carbons from Pine Wastes | High benzene adsorption rates | Garcia-Garcia et al.,2002 |
| Rice Husk Ash of Kenyan Origin | Phenolic Compounds | | Mbui et al., 2002 |
| Low-Molecular-Weight Polyacrylic Acid | Silica, Alumina, and Kaolin | | Zaman et al., 2002 |
| Cement Kiln Dust | Cationic Dyes | | Nassar et al., 2002 |
| Sludge Ash | New Coccine Dye | The ash adsorption capacities for the dye were in the range 3.25-5.70 mol/g and were affected by the pH, ionic strength and temperature | Chih-Huang Weng, 2002 |
| Bone-like Carbonated Calcium Phosphates | Aminoacids | | Bihi et al., 2002 |
| Aluminum-Containing Mesoporous Silica Films | Methylene Blue | Methylene blue was adsorbed effectively on the mesoporous silica films from solutions. | Yoshikawa et al., 2002 |
| Calcined Alunite and Granular Activated Carbon | Acid Dyes | | Özacar and Sengil, 2002 |

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| Low-Cost Material<br><br>18/18 | Victoria Blue | victoria blue is physically adsorbed onto the perlite | Demirbas et al., 2002 |
| Chitosan-encapsulated Activated Carbon | Dyes and Humic Acid | | Wu et al., 2002 |
| Activated Carbon | Acid Dyes | Surface diffusivity may vary as a function of surface coverage. | Ko et al., 2002 |
| Activated Carbon | p-Cresol & p-Nitrophenol | | Nouri et al., 2002 |
| Metal(hydr)oxides | Simple Organic Acids | | G. Horányi 2002 |
| Activated Carbon | organic contaminants | | Schmotzer et al., 2002 |
| HDTMA Modified Kaolinite and Halloysite | Naphthalene | | Lee et al., 2002 |
| Activated Carbon | Aromatic Compound | | Haghseresht et al.,2002 |
| Novel Chitosan Gel Modified by Phenylboronate | Glucose | | Matsumoto et al., 2002 |
| Montmorillonite-based Filler Clay | Fatty Acids | | Khalil et al., 2002 |
| $TiO_2$ Photocatalysis | 2-chlorophenol | | Ilisz et al., 2002 |
| Activated Carbon | Dissolved Organic Matter | | Li et al., 2002 |

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| Activated Carbon Fibres | Trace Volatile Organic Compounds | ACFs with specific surface areas of 640 m2/g and 1460 m2/g were used to adsorb trace volatile organic compounds in nitrogen streams at atmospheric pressure at or near room temperature (25°C and 30°C). | Huang et al., 2002 |
| Granular Activated Carbon | Chlorobenzene | high-capacity adsorption on GAC up to about 450 mg per gram. | Lorbeer et al., 2002 |
| Live and Dead Microalgal Cells | Tributyltin (TBT) | | Tam et al., 2002 |
| Spherical Activated Carbons from Oil Agglomerated Bituminous Coals | Organic Impurities | | Gryglewicz et al., 2002 |
| Granulated Iron Hydroxide | Reactive Dyes | | Kornmuller et al., 2002 |
| Sepiolite | Quaternary Amines | | Sabah and Çelik, 2002 |
| Acid- and Heat-activated Sepiolites | Cationic Surfactants | | Sabah et al., 2002 |
| Amidoximated Cellulose | Dyes | | Saliba et al., 2002 |
| Activated Carbon | Organic Contaminants | | Schmotzer et al., 2002 |
| Activated Carbons | Aromatic Compounds | | Haghseresht et al., 2002 |

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| Carbonaceous Materials from Wood Chips | Bisphenol A | | Nakanishi et al., 2002 |
| Toluene | Asphaltenes | | Marczewski and Szymula, 2002 |
| Bentonite Clay | Hemoglobin | | Bajpai and Sachdeva, 2002 |
| Granular Activated Carbon | Pesticide | | Matsui et al., 2002 |
| Treated Cotton | Acid Dyes | | Bouzaida and Rammah, 2002 |
| 1,1'-Dimethyl-4,4'-bipyridinium-smectites | Phenols | The increase in the basal spacing by the reaction with phenols indicates a change in the interlayer microstructure. | Okada and Ogawa, 2002 |
| Activated Carbons | Olive Mill Waste | | Galiatsatou et al., 2002 |
| Carbon Blacks | Ionic Surfactant | | González-García e al.,2002 |
| Poly(trimethylene Terephthalate) | Disperse Dyes | | Yang et al., 2002 |
| Eucalyptus Barks | Dyes | | Saliba et al.,2002 |
| Wheat Straw, Corncobs and Barley Husks | Dye | | Robinson et al., 2002 |
| GAC | Lindane | | Sotelo et al.,2002 |
| Chitosan | Blood Protein | | Benesch and Tengvall, 2002 |
| Bagasse Fly Ash | Lindane and Malathion | | Gupta et al., 2002 |

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| Activated Carbon | Phenol | | Polaert et al.,2002 |
| Activated Carbon Preloaded with Humic Substances | Trichloroethylene | | Kilduff and Karanfil, 2002 |
| Corncob and Barley Husk | Artificial Textile Dye Effluent | | Robinson et al., 2002 |
| Bentonite and Perlite | P-chlorophenol | | Koumanova and Peeva-Antova , 2002 |
| Graft Copolymers of Sawdust/Vinyl Monomers | Dye | | Wang et al., 2002 |
| Goethite and Kaolinite | Anthracene | | Angove et al., 2002 |
| Carbons | Paracetamol | | Artur P. Terzyk, 2002 |
| Activated Carbon | Aromatic Compounds | | Haghseresht et al., 2002 |
| Coal Based Sorbents | Direct azo Dye | | Mohan et al., 2002 |
| Bone-like Carbonated Calcium Phosphates | Amino acids | | Bihi et al., 2002 |
| Kaolinite and Montmorillonite | Quinoline | | Burgos et al., 2002 |
| Organobentonite | Phenol | | Yun-Hwei Shen, 2002 |
| Activated Carbon | Aromatic Compounds | | Nouri et al., 2002 |
| Modified Cotton | Dye | | Khalfaoui et al., 2002 |

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| Modified Cotton | Dye | | Khalfaoui et al., 2002 |
| Montmorillonites | Dinitrophenol | | Sheng et al., 2002 |
| *Rhizomucor pusillus* | Colour from a Bleach Plant Effluent | | Christov and Driessel, 2002 |
| Activated Carbon | Nitrophenol | | Chern and Chien, 2002 |
| Sepiolite | Diquat, Paraquat and Methyl Green | | Rytwo et al., 2002 |
| Kaolinite | Methylene Blue | | Ghosh and Bhattacharyya,2002 |
| Rubber Seed Coat | Phenol | | Rengaraj et al,2002 |
| Zirconium Pillared Clay | Tannic Acid | | Anirudhan and Vinod, 2002 |
| Activated Carbon | Benzene and Methylethylketone | | Huang et al., 2002 |
| Rice Bran | Chloroform | | Adachi et al., 2002 |
| Silica supported cyclodextrin Derivatives | Organic Pollutants | | Phan et al., 2002 |
| Activated Carbon Fiber | Dye | | Ko et al., 2002 |
| Paper | Dyes | | Wang et al., 2002 |
| 44/45 | | | |
| Treated Cotton | Acid Dyes | | Bouzaida and Rammah, 2002 |
| High Surface Activated Charcoal | Acetaminophen (Paracetamol) | | Hoegberg et al., 2002 |

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| Carbon Blacks | Ionic Surfactant | | González-García et al., 2002 |
| Granulated Iron Hydroxide | Reactive Dyes | | Kornmüller et al., 2002 |
| Bentonite Clays | Polyethyleneimine | | Öztekin et al., 2002 |
| Activated Carbons Produced from Waste Tyre Rubber | Organic Compounds | | Miguel et al., 2002 |
| Low-cost Adsorbent | Anionic Surfactant | | Purakayastha et al., 2002 |
| Activated Carbon - | Acetaminophen | | Nakamura et al., 2002 |
| Chemically Modified Chitosan Beads | Indomethacin | | Mi et al., 2002 |
| Activated Carbon Immobilized with *Pseudomonas putida* | Phenol | | Annadurai et al., 2002 |
| Activated Sludge Biomass | Basic Dyes | | Chu and Chen, 2002 |
| Activated Carbons | Phenol | | Fernandez et al., 2003 |
| Pecan Shell and Almond Shell based Granular Activated Carbons | Volatile Organic Compounds | | Bansode et al., 2003 |
| Perlite | Methyl Violet | | Doan and Alkan, 2003 |
| Coal | Sodium Dodecyl Benzenesulfonate | | Mishra et al., 2003 |

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| Diatomaceous Earth | Dyes from Textile Wastewater | | Al-Ghouti et al., 2003 |
| Activated Clays | Dyes and Surfactants | | Espantaleón et al., 2003 |
| Diatomaceous Clay | Basic Dye | | Shawabkeh and Tutunji, 2003 |
| Activated Carbon | 2-Naphthalene sulfonate | | Chang et al., 2003 |
| Natural Clinoptilolite | 1, 2-Dichloroethane | | Pilchowski and Chmielewská, 2003 |
| Activated Carbon from Peanut Shells | Organic Compounds | A useful correlation for the calculation of the affinity coefficient as a function of relative parachor is presented. | E. Elio Gonzo, 2003 |
| Bituminous Shale | Basic Blue 41 Dye | It was found that the smaller the particle size, the higher the adsorption. The adsorption capacity increased with temperature. | Müftüoglu et al.,2003 |
| Activated Carbon | VOCs | | Chuang et al., 2003 |
| Activated Carbons | Chlorinated Organic Compounds | | Bembnowska et al., 2003 |
| Chitosan | Acid Dye | | Wong et al., 2003 |
| Silica Modified with Humic Acids | Indigo Carmine Dye | | Prado et al., 2003 |
| Low-cost Adsorbents | Basic Dye (basic Red) | | Gupta et al., 2003 |

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| Activated Bentonites | Phenol | | Al-Asheh et al., 2003 |
| Activated Carbon | Chloroacetanilide Herbicides | | Gustafson et al., 2003 |
| Natural and Modified Zeolites | Azo Dyes | | Armaan et al., 2003 |
| Activated Carbons | Phenol | | Salame and Bandosz, 2003 |
| Activated Carbons from Date Pits Impregnated with Potassium Hydroxide | Methylene Blue | The adsorption capacity increased from 80.3 mg g to 123.1 mg g upon chemical activation. | Banat et al., 2003 |
| Adsorbent Materials from Sewage Sludges | Methylene Blue | | Otero et al., 2003 |
| Granular Activated Carbon | Volatile Organic Compounds | | Aizpuru et al., 2003 |
| Polymer-Based Active Carbons | Phenol and 2,3,4-Trichlorophenol | | László et al., 2003 |
| Activated Carbons from Surplus Sewage Sludge | Dyes | | Martin et al., 2003 |
| Soil | Triasulfuron | | Pusino et al., 2003 |
| White rot fungus (*Fomes lividus*) | azo dyes and dye industry effluent | In batch mode treatment, a maximum decolorization of 84.4% was achieved on day 4, and in continuous mode a maximum decolorization of 37.5% was obtained on day 5. | Selvam and Swaminathan, 2003 |

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| Surfactant-Modified Sepiolite | Negatively Charged Azo Dyes | | Armagan et al., 2003 |
| Diatomaceous Material | Acetaldehyde | | Kanno et al., 2003 |
| High-Silica Zeolites | VOC | | Monneyron et al., 2003 |
| Activated Carbon | Phenolic Compounds | the capacity of the activated carbon used to adsorb these compounds presented the following order: syringic acid > *p*-hydroxybenzoic acid > gallic acid | García-Araya et al., 2003 |
| Shale Oil Ash | Dyes | | Al-Qodah and Lafi, 2003 |
| Neem Leaf Powder | Brilliant Green Dye | | Bhattacharyya and Sarma, 2003 |
| pyrolyzed oil shale residue | phenol | Chemically activated oil shale, pretreated with $ZnCl_2$, gave the highest uptake of phenol. | Al-Asheh et al., 2003 |
| Low-cost Coir Pith Carbon | 2-chlorophenol | | Namasivayam and Kavitha, 2003 |
| Activated Carbon | Carbon Tetrachloride and Chloroform | | Shim et al., 2003 |
| Granular Activated Carbons | Acetone, Methyl Ethyl Ketone, 1,1,1-Trichloroethane, and Trichloroethylene | | Pires et al., 2003 |
| Dolomitic Sorbents | Dye | | Walker et al., 2003 |

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| Kaolinite | Poly(vinylpyrrolidone) and Sodium Dodecylbenzenesulfonate | | Torn et al., 2003 |
| Tobermorite | Natural Organic Polyelectrolytes | | Kaneco et al., 2003 |
| Activated Carbon | Benzoic Acid and P-nitrophenol | | Chern and Chien, 2003 |
| Activated Carbon from Apricot Stones | p-chlorophenol | | Koumanova et al., 2003 |
| Iron Oxides | Residual Organic Matter | | Choo and Kang, 2003 |
| Activated Sludge | Basic Dye (maxilon Red BL-N) | | Basibuyukand Forster, 2003 |
| Fuller's Earth | Methylene Blue | | Atun et al., 2003 |
| Rayon-based Activated Carbon Fibers | Formaldehyde | | Rong et al., 2003 |
| Bentonites Modified with Single or Dual Quaternary Ammonium Cations | Organic Compounds | the adsorption affinity on dual-modified bentonites was generally lower than that on single-modified bentonites | Gönülşen et al., 2003 |
| Activated and Non-activated Oak Shells | Phenol and Dyes | Interaction amongst the different operating variables played a role in the uptake of phenol or Methylene Blue dye by the adsorbent considered. | Al-Asheh et al., 2003 |

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| Clinoptilolite | Cationic Surfactants | | Ersoy and Çelik, 2003 |
| Chemical Cross-linked Chitosan Beads | Dye | | Chiou and Li, 2003 |
| Rice Husk-based Active Carbon | Malachite Green | | Guo et al., 2003 |
| Activated Carbons from Sawdust and Rice-husk | Acid Yellow 36 | | P. K. Malik, 2003 |
| Heat Treated Kerolites | Atrazine | | González-Pradas et al.,2003 |
| Modified Peat–resin Particle | Basic Dyes | | Sun and Yang, 2003 |
| Activated Carbon Cloths | Dyes | | Métivier-Pignon et al.,2003 |
| Sewage Sludge | Organic Water Pollutants | | Otero et al., 2003 |
| Apple Pomace | Pectin and Phenolic Compounds | | Schieber et al., 2003 |
| Powdered Activated Charcoal | Ammonium Dinitramide (ADN) | | Santhosh et al., 2003 |
| Calcined Alunite | Dyes | | Özacar and Sengil 2003 |
| Pine Bark | Lindane and Heptachlor | | Ratola et al., 2003 |
| Poly(acrylamide-co-acrylic Acid) Hydrogels | Methyl Violet | | Solpan et al., 2003 |
| Waste Metal Hydroxide Sludge | Azo Reactive Dyes | | Netpradit et al., 2003 |

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| Activated Carbon Fibre-based Monoliths | Volatile Organic Compounds | | Fuertes et al., 2003 |
| Adsorbent from Elutrilithe | MCPA (4-Chloro-2-Methylphenoxy-Acetic Acid) | | Mehmet Mahramanlioglu, 2003 |
| Pine Bark | Lindane | Excellent efficiency of adsorption (average 80.65%) | Ratola and Botelho, 2003 |
| Limed Forest Soil | Low Molecular Weight Organic Acids | | Van Hees and Jones, 2003 |
| Cholesterol Surface | Dyes (Methylene Blue and Acridine Orange and Their Mixtures) | The adsorption process was controlled by the ion-dipole and hydrophobic interactions. | D. Palit, 2003 |
| Organomont--morillonites | Toluene Vapors | | Muminov and Gulyamova, 2003 |
| Granular Activated Carbon | Resorcinol and Catechol | | Kumar et al., 2003 |
| Activated Carbon | Olive Mill Effluent Wastewater | | Azzam et al., 2003 |
| Soils | Triazine Herbicides | | Yang et al., 2003 |
| Perlite | Methyl Violet | | Dogan and Alkan 2003 |
| Activated Carbons | Polycyclic Aromatic Hydrocarbon | | Mastral et al., 2003 |
| Synthetic Rubber | Ethoxylated Nonyl Phenol | | El-Feky and Shalaby, 2003 |
| Mesoscopic Mica Flakes | Cyanine Dyes | | Karthaus and Kawatani, 2003 |

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| Mesoporous Silica | Proteins | | Deere et al., 2003 |
| Chitosan | Acid Dyes | | Wong et al., 2004 |
| Aminosilane Modified $TiO_2$ Surface | Organic Dyes | | Andrzejewska et al., 2004 |
| Prosopis cineraria an Agro-industry Waste | Malachite Green Dye | | Garg et al., 2004 |
| Mezoporous Minerals | Azo-reactive Dyes | | Ozdemir et al., 2004 |
| Balkaya Lignite | Methylene Blue | | Karaca et al., 2004 |
| Novel Mesoporous Carbons | Inflammatory Cytokine | | Malik et al., 2004 |
| Activated Carbon | Phenolics | | Lu and Sorial, 2004 |
| Activated Bleaching Earth | Herbicide Paraquat | | Tsai et al., 2004 |
| Silica Adsorbents | Amino Acids, Small Peptides, and Nucleic Acid | | Vladimir A. Basiuk, 2004 |
| Dry Soil | Volatile Chlorinated Organic Compounds | | Kobayashi et al., 2004 |
| Polyacrylic Acid-bound Iron Oxide Magnetic Nanoparticles | Methylene Blue | | Mak and Chen, 2004 |
| Activated Carbon | 2-mercaptothiazoline | | Chang et al., 2004 |
| ion-exchange resins | Organic acids | | Gluszcz et al., 2004 |

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|---|---|---|---|
| Alunite | disperse dyes | | Özacar and Şengil, 2004 |
| Amphoteric Ion Exchange Fibers | Amino Acids | | Fu et al., 2004 |
| Cation-exchanged Montmorillonites | Valine | | Nagy and Kónya, 2004 |
| Activated Carbons Derived from Solid Wastes | Phenol and Reactive Dye | | Nakagawa et al., 2004 |
| Silica | Vitamin E | | Chu et al., 2004 |
| Peat and Soil Samples | Organophosphorus Pesticides | | Rotich et al., 2004 |
| Chitosan | Acid Dyes | | Wong et al., 2004 |
| Metal Hydroxide Sludge | Azo Reactive Dyes | | Netpradit et al., 2004 |
| Methylated Yeast Biomass | Egg Albumin | | Seki et al., 2004 |
| Polyurethane-chitosan Blends | Color Dyestuffs | | Shih et al., 2004 |
| Silicon Oxide-Aqueous Solution Interface | Casein | | Follows et al., 2004 |
| Granular Activated Carbon | Phenolphthalein | | Tansel and Nagarajan, 2004 |
| $CO_2$LaserModified Magnesia Partially Stabilised Zirconia (MgO-PSZ) | Human Serum Albumin (HSA) | | Hao and Lawrence, 2004 |
| Polymeric Adsorbents | Phenol | | Li et al., 2004 |

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| Activated Carbon.I | Volatile Organic Compound | | Lordgooei and Kim, 2004 |
| Activated Carbon.II | Volatile Organic Compound | | Lordgooei and Kim, 2004 |
| Phenol-, Tire-, and Coal-Derived Activated Carbons | Organic Vapors | | Ramirez et al., 2004 |
| Cationized Solid Wood Residues | Orthophosphate | | Karthikeyan et al., 2004 |
| PAC | Organic Matter | | Tomaszewska et al., 2004 |
| Silica Surface: 1 | Cationic Surfactants | | Golub and Koopal, 2004 |
| Silica Surface: 2 | Cationic Surfactants | | T. P. Golub, 2004 |
| Quartz Sand | Extracellular Glucose Oxidase from *Penicillium funiculosum* 46.1 | | Eryomin et al., 2004 |
| Untreated and Treated Activated Carbon | *p*-Nitrophenol | | SirousNouri, 2004 |
| Hydrotalcite | 2,4-Dichlorophenol | | Yapar et al., 2004 |
| Organoclays | Phenanthrene | | El-Nahhal and Safi, 2004 |
| Clay/water System | Methylene Blue | | Gürses et al., 2004 |
| Bentonites | Poly (4-vinylpyridine) | | Bacquet et al., 2004 |

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| Ozone-adsorbed High Silica Zeolites | Trichloroethene | | Fujita et al., 2004 |
| Electro-activated Carbon Granules | Phenol | | Lounici et al., 2004 |
| Montmorillonite Modified with Natural Organic Cations | Herbicide Simazine | | Cruz-Guzmán et al., 2004 |
| Modified Resin | Water-soluble Dyes | | Yu et al., 2004 |
| Alumina Interface | Sucrose | | Singh and Mohan 2004 |
| Raw and Activated Date Pits | Phenol | | Banat et al., 2004 |
| Carbon Materials | Organic Molecules | | Carlos Moreno-Castilla,2004 |
| Natural Clay Minerals with Hexadecylpyridinium Cation | Fungicides | | Andrades et al., 2004 |
| Cross-linked Chitosan Beads | Anionic Dyes | | Chiou et al., 2004 |
| Kaolinite | Phenoxy acetic acid Herbicides | | Tunega et al., 2004 |
| Activated Carbon | Phenolic Compounds | | Dabrowski et al., 2005 |
| Lysozyme Crystals | Xanthene Dyes | | Cvetkovic et al., 2005 |
| Zeolite | Naphthalene | | Yuh-ShaHo, 2005 |
| Diatomite Earth | Textile Dyes | | Erdem et al., 2005 |
| Alumina | Anionic Surfactant | | Adak et al., 2005 |

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# CHAPTER- 3

# Adsorption of Copper from aqueous solution on *Brassica cumpestris* (Mustard Oil Cake)

# Adsorption of Copper from aqueous solution on *Brassica cumpestris* (Mustard oil cake)

## 3.1.Introduction

Industrialization has a tremendous impact on the concentration and distribution of heavy metals in the atmosphere, on the land and in the water bodies. The extent of this widespread but generally diffuse contamination has caused concern about its possible effects on the plants, animals & human beings. Among these contaminants copper is a metal of concern. Copper is generally considered to be non-toxic for man but at concentration exceeding 5 mg $l^{-1}$ impart color and undesirable taste to water. The World Health Organization's guideline for drinking water based on its staining properties is 1mg $l^{-1}$[1].Beyond the permissible level (5 mg $l^{-1}$) copper causes acute and chronic disorders in human beings such as gastrointestinal catarrh, cramps in the calves, hemochomatosis and skin dermatitis brasschills, usually accompanied by high fever [2,3]. Industries discharging copper in the wastewater are electroplating industries, pulp and paper mills, fertilizer plants, steel work foundries, petroleum refineries, aircraft plating and finishing, motor vehicles, non-ferrous metal works [4-6].

Processes generally used for the removal of Cu (II) from wastewater include precipitation, evaporation, reverse osmosis and ion exchange. But most of these process are economically non feasible for small scale industries to treat the waste effluents. The adsorption process has been found to be economically appealing for the removal of heavy metals from wastewater. For the treatment of copper rich effluents at the solid-solution interface several adsorbents have been used earlier [7-13]. The adsorption behavior of fruit feel of orange, sawdust, kyanite and rice husk for the removal and recovery of Hg(II), Cr(VI), Ni(II), Cu(II), Cd(II), Pb(II), Zn(II) and Mg(II) from rivers and industrial wastewater have been studied in our laboratory[14-18].

In the present study a new adsorbent mustard oil cake (MOC) prepared from the seeds of Brassica cumpestris (mustard) is used. It is a valuable by-product left after the extraction of oil is known as mustard oil cake (MOC). It contains significant amount of proteins, of value as animal feed if edible and as nitrogenous manures otherwise. The chemical composition of mustard oil cake shows: nitrogen-4.5% and phosphorous penta oxide – 1.5% [19].

## 3.2-Material and Method:

### 3.2,1.Adsorbent:

Oil is extracted from the *Brassica cumpestris* (mustard) seeds and waste matter left after extraction is known as mustard oil cake (MOC). The mustard oil cake treated with hot double distilled water in order to remove the oil and finally dried in an air over at 60-65°C for 24 h. After drying the adsorbent was sieved through 40-60 mesh size (BSS) and used as such.

### 3.2,2.Adsorbate solution:

Stock solution of Cu (II) was prepared (1000 mg $l^{-1}$) by dissolving the desired quantity of Copper nitrate trihydrate (AR grade) in distilled water. Solutions of other metal ions were prepared (1000 mg $l^{-1}$) by dissolving their chlorides or nitrates.

### 3.2,3.Adsorption studies:

Adsorption studies were carried out by batch process. 0.5 g adsorbent was placed in a conical flask in which 50 ml solution of metal ion of desired concentration was added and the mixture was shaken in shaker. The mixture was then filtered and final concentration of metal ion was determined in the filtrate by atomic absorption spectrophotometer (GBC 902). The amount of metal ions adsorbed was calculated by subtracting final concentration from initial concentration. All the experiments were carried out in triplicate and mean concentration was calculated by averaging them. The relative standard deviation (R.S.D.) for each sample was calculated. The values of R.S.D. (on percent basis) in all the experiments were found to be in the range 0.548%-1.252%.

### 3.2,4.Effect of pH:

The effect of pH on the adsorption of Cu (II) was studied as follows:

100 ml of Cu (II) solution was taken in a beaker. The pH of solution was adjusted by adding dilute solution of hydrochloric acid or sodium hydroxide. The concentration of Cu (II) in this solution was then determined (initial concentration). 50

ml of this solution was taken in a conical flask and treated with 0.5 g of adsorbent and after equilibrium the final concentration of Cu (II) was determined.

**3.2,5.Effect of time:**

A series of 250 ml conical flask, each having 0.5 g adsorbent and 50 ml solution (of known Cu (II) concentrations) were shaken in a shaker incubator and at the predetermined intervals the solution of the specified flask was taken out and filtered. The concentration of Cu (II) in the filtrate was determined by atomic absorption spectrophotometer (GBC 902 model). The amount of Cu (II) adsorbed in each case was then determined as described earlier.

**3.2,6.Effect adsorbent dose:** ]

A series of 250 ml conical flasks each containing 50 ml of Cu (II) solution of 50 mg $l^{-1}$ concentration were treated at $20^0$C with varying amount of adsorbent (0.1 to 1.0 g) at pH 4. The flasks were shaken in a shaker incubator and after equilibrium the solutions were filtered .The amount of Cu (II) in the filtrate was then determined by atomic absorption spectrophotometer. The amount of Cu (II) adsorbed in each case was then calculated as described above. The same procedure was repeated at 30 and $40^0$C.

**3.2,7.Breakthrough Capacity:**

0.5 g of adsorbent was taken in a glass column (0.6 cm internal diameter) with glass wool support. One liter of Cu (II) solution with 50 mg $l^{-1}$initial concentrations ($C_0$) was then passed through the column with a flow rate of 1ml. min $^{-1}$. The effluent was collected in 50 ml fractions and the amount of Cu (II), (C) was determined in each fraction with the help of atomic absorption spectrophotometer. The breakthrough curve was obtained by plotting C/Co Vs volume of the effluent.

**3.2,8. Desorption Studies:**

Desorption of Cu (II) was carried out as follows:

0.5 g of adsorbent was treated with 50 ml Cu (II) solution (50 mg $l^{-1}$) in a conical flask. The solution was filtered after 24 h. The adsorbent was then washed several times with distilled water to remove any excess of Cu (II). It was then treated with 50 ml of 0.1 M sodium chloride solution and then filtered after 24 h. The filtrate was analyzed for Cu (II) desorbed. The same procedure was repeated with potassium sulphate and hydrochloric acid solutions.

**3.2,9. Regeneration Studies:**

0.5 g of adsorbent was treated with 50 ml Cu (II) solution (50 mg. $l^{-1}$) in a conical flask and after equilibrium it was filtered. The adsorbent was then treated with 50 ml hydrochloric acid solution (0.05 M) for 24 h. It was filtered and filtrate was analyzed for Cu (II) desorbed. The adsorbent was washed several times with distilled water in order to remove excess acid. It was again treated with 50 ml of Cu (II) solution and the above procedure was repeated a number of times (five times or cycles). The same procedure was repeated with 0.1 M hydrochloric acid solution

## 3.3. Result and discussion:

The adsorption behavior of different metal ions on mustard oil cake (MOC) is shown in Table 3.1.The adsorption or metal uptake (mmols $g^{-1}$) of Cu (II) was found to be maximum followed by Zn (II), Cr (VI), Mn (II), Cd (II), Ni (II) and Pb (II).

### 3.3,1. Effect of time and initial concentration:

The effect of time and amount adsorbed (x/m) by MOC is presented in Figure-1.The adsorption of Cu (II) has been shown to increase with time and attains a maximum value at 20 minutes and then remains almost constant for 50 mg $l^{-1}$ initial Cu (II) concentration, but below this concentration the maximum adsorption occurs much earlier. When initial Cu (II) concentration is increased from 5-mg. $l^{-1}$ to 50-mg. $l^{-1}$, the amount adsorbed increase from 8.8-to 96 mg $l^{-1}$ showing that adsorption of Cu (II) depends upon the initial concentration because amount of Cu (II) adsorbed increases by increasing the initial concentration.

### 3.3,2. Effect of pH:

The effect of pH on the adsorption of Cu (II) on MOC in shown in Figure-2. The percentage adsorption varies from 44% to 94%. When pH is increased from 2 to 10 at 50 mg $l^{-1}$ initial concentrations. The maximum percentage adsorption is observed between pH 3-4. At this pH there are two species of Cu (II) present in the solution [15].

(i) Cu (II) (large quantity)

(ii) Cu OH (small quantity)

The maximum adsorption at pH 3-4 indicates that Cu (II) ions are predominantly adsorbed on MOC (since Cu (II) ions are present in large quantity in this pH range) either by ion exchange or by hydrogen bonding. The % adsorption increases slowly with increasing pH and becomes constant above pH 6 due to the precipitation of Cu (II) as Cu $(OH)_2$.

**3.3,3. Adsorption isotherms:**

The adsorption isotherm data were analyzed with Langmuir and Freundlich isotherms [14]. The values of θ and b were calculated from the slope and intercept of the linear plots of 1/ (x/m) Vs 1/Ce (Table 3.2).

This essential feature of the Langmuir isotherm can be expressed in terms of dimensionless constant separation or equilibrium parameter ($R_L$), which is defined as:

$$R_L = 1/ 1+bC_o \qquad \text{\_\_\_\_\_\_\_\_(1)}$$

Where $C_o$ is the initial metal ion concentration (mg $l^{-1}$) and b is the Langmuir constant. The values of $R_L$ reported in Table 2 at different temperatures are less than unity ($R_L$ <1) showing favorable adsorption of Cu (II) on MOC [20]. The values of $K_f$ and n were calculated from the intercept and slope of the Freundlich plots. These values are reported in Table 2.The values of n between 1 and 10 represent beneficial adsorption [21].

**3.3,4. Effect of temperature**:

The temperature range used in this study was 20 to 40 °C.The values of equilibrium constants ($K_c$) at 20,30 and 40°C were calculated from the following relation [22].

$$Kc = C_{Ac} / C_e \qquad \text{\_\_\_\_\_\_\_\_\_\_(2)}$$

Where $C_{Ac}$ and $C_e$ are the equilibrium concentrations (mg. $l^{-1}$) of Cu (II) on the adsorbent and in solution respectively.

$$\Delta G^o = - RT \ln Kc \qquad \text{\_\_\_\_\_\_\_\_(3)}$$

Where T is the absolute temperature, R is gas constant and $\Delta G^o$ is the standard free energy change. The values of enthalpy change ($\Delta H^o$) and entropy change ($\Delta S^o$) were calculated from the following relation:

$$\log Kc = \Delta S^o / 2.303R - \Delta H^o / 2.303RT \qquad \text{\_\_\_\_\_\_\_\_\_\_}(4)$$

$\Delta S^o$ and $\Delta H^o$ were calculated from the slope and intercept of linear plot of log $K_c$ Vs 1/T (Figure 3). Table 3.3 shows the values of Kc, $\Delta H^o$, $\Delta S^o$ and $\Delta G^o$. The positive value of $\Delta H^o$ indicates that the process is endothermic. The negative values of $\Delta G^o$ show that process is spontaneous and spontaneity increases with increase in temperature .The positive value of $\Delta S^o$ suggests an increase randomness at the solid –liquid interface during adsorption.

**3.3,5. Adsorption kinetics:**

The rate constants were calculated by using Lagergren first order and pseudo-second order kinetic equations. Lagergren first order expression is given by equation:

$$\log (qe-q) = \text{Log}\ qe - K_1/2.303\ .t \qquad \text{\_\_\_\_\_\_\_\_\_\_}(5)$$

Where qe is the amount adsorbed at equilibrium, q is the amount adsorbed at time t and K1 is the adsorption rate constant. The linear plot of log (qe-q) Vs t is observed (Figure 4). The regression coefficient ($R^2$) in this case is 0.9971.The value of rate constant $K_1$ as determined from the slope of the line was 0.357 $min^{-1}$ the pseudo second order adsorption kinetic rate equation is given as

$$t/q = 1/K_2qe^2 + 1/qe.t \qquad \text{\_\_\_\_\_\_\_\_\_\_}(6)$$

Where $K_2$ is the adsorption rate constant of pseudo-second order kinetics(g $mg^{-1}min^{-1}$). The value of $K_2$ was calculated from the slope of the linear plot of t/q Vs t

(Figure: 5). A comparison of the experimental sorption capacities and calculated values obtained from equation 5 and 6 shows that qe (theor) value calculated from the pseudo-first order kinetic model (42.6 mg. $g^{-1}$) differed largely from qe (exp) value (48.0 mg $g^{-1}$). In pseudo second-order kinetics the calculated qe (theor) value (48.0 mg $g^{-1}$) was found to be very close to qe(exp) value (48.0 mg $g^{-1}$) and also the high value (0.9998) of the correlation coefficient ($R^2$) confirmed the applicability of the pseudo-second order kinetics.

**3.3,6. Breakthrough Capacity**:

The breakthrough curve for 50 mg $l^{-1}$ initial Cu (II) concentration and a flow rate of 1 ml $min^{-1}$ with 0.5 g adsorbent is shown in Fig. 6. The breakthrough curve showed that 50 ml of the solution (corresponding to 2.5 mg Cu (II)) could be passed through the column without detecting Cu (II) in the effluent. The breakthrough and exhaustive capacities were determined as 5 and 10 mg $g-^1$ respectively.

**3.3,7. Desorption studies**:

In order to make the adsorption process more economical it is important to desorb and regenerate the spent adsorbent. The desorption studies were carried out by batch process using NaCl, $K_2SO_4$ and HCl solutions of different concentrations. The results are reported in Table 3.4. Desorption of Cu (II) with NaCl or $K_2SO_4$ is negligible showing that Cu (II) is strongly adsorbed on MOC.This is important because Cu (II) adsorbed by MOC will not be exchanged by NaCl or $K_2SO_4$ if MOC is to be used to sequester Cu ions in soil since appreciable amount of these salts are usually present in the soil. However, desorption up to the extent of 67% could be achieved when 0.1 M hydrochloric acid solution was used as eluent. Desorption of metal ions with acid solution indicates that adsorption of Cu (II) is via. Ion- exchange process.

Attempts were made to regenerate the spent adsorbent with 0.05 M and 0.1 M hydrochloric acid solutions. The results were almost similar. Fig. 7 shows that % adsorption decreases from 86 to 64% after first regeneration and recovery of Cu (II) is 67.4%. The % adsorption then remains almost constant (64 - 60%) in the subsequent cycles. The remarkable decreases in the first regeneration cycle indicates that perhaps certain adsorption sites or functional groups are decomposed or destroyed by hydrochloric acid and hence it is expected that Cu (II) is weakly adsorbed in the second, third and fourth cycle. However, 60% adsorption could be achieved up to fourth regeneration cycle.

## 3.4- Conclusion:

The mustard oil cake (MOC) is a low cost material abundantly available in India. It is used as manure in agriculture to provide nitrogen and phosphorous (essential plant nutrients). Its adsorption properties could be utilized to sequester Cu (II) ions in the soil. The order of adsorption of various heavy metals on MOC is Cu (II) > Zn (II) > Cr (VI) >Mn (II) > Cd (II) > Ni (II) > Pb (II).

The adsorption of Cu (II) is pH dependent and maximum adsorption occurs at pH 3 - 4. The adsorption follows pseudo-first and pseudo-second order kinetic but pseudo – second order kinetic equation is better obeyed. The adsorption process is endothermic and spontaneous in nature. Breakthrough capacity shows that 50 ml of water (corresponding to 2.5 mg of Cu (II) ) can be treated without detecting Cu (II) in the effluent. The Cu (II) ions are strongly adsorbed on MOC and cannot be recovered with various neutral salt solutions like NaCl, $K_2SO_4$ etc. The recovery of Cu (II) can be made to some extent with dilute hydrochloric acid solution but treatment of MOC with hydrochloric acid remarkably reduces the capacity of adsorbent.

**Table3.1.** Adsorption of different metal ions on to Mustard oil cake (MOC)

| S No. | Metal ions | Adsorption (mmolsgm-[1]) |
|---|---|---|
| 1 | Zn (II) | 0.691 |
| 2 | Mn (II) | 0.582 |
| 3 | Cu (II) | 0.717 |
| 4 | Cr (VI) | 0.675 |
| 5 | Cd (II) | 0.396 |
| 6 | Pb (II) | 0.183 |
| 7 | Ni (II) | 0.357 |

**Table 3.2.** Langmuir and Freundlich constants at different temperature for the adsorption of Cu (II) on to MOC

| Temperature ($^0$C) | Langmuir isotherms | | | | | Freundlich isotherms | | | |
|---|---|---|---|---|---|---|---|---|---|
| | $\theta^{\circ}.b$ ($lg^{-1}$) | $\theta^{\circ}$ ($mgg^{-1}$) | b ($lmg^{-1}$) | $R^2$ | $R_L$ | Log $K_f$ | $K_f$ | n | $R^2$ |
| 20 | 63.350 | 454.54 | 0.139 | 0.9901 | 0.887 | 1.8512 | 70.99 | 1.82 | 0.9933 |
| 30 | 78.125 | 434.78 | 0.179 | 0.9842 | 0.848 | 1.9515 | 89.43 | 2.09 | 0.9988 |
| 40 | 74.620 | 714.28 | 0.104 | 0.9853 | 0.905 | 1.9539 | 89.92 | 1.69 | 0.9692 |

**Table 3.3**. Thermodynamics parameters at different temperature for the adsorption of Cu (II) on MOC

| Temperature (° C) | $K_C$ | $\Delta G^\circ$ (kJ $mol^{-1}$) | $\Delta H^\circ$ (kJ $mol^{-1}$) | $\Delta S^\circ$ (kJ $mol^{-1}$) |
|---|---|---|---|---|
| 20 | 12.88 | - 6.247 | | |
| 30 | 15.13 | - 6.842 | 11.105 | 0.059 |
| 40 | 16.85 | - 7.337 | | |

**Table 3.4.** Desorption of Cu (II) by various eluents

| S No. | Amount of Cu (II) adsorbed(mg)/0.5g adsorbent | Amount of Cu (II) recovered (mg) | % Recovery | Eluent used |
|---|---|---|---|---|
| 1 | 45 | 1.1 | 2.2 | 0.1M NaCl |
| 2 | 42.8 | 1.2 | 2.8 | 1.0M NaCl |
| 3 | 42.1 | 2.0 | 4.0 | 0.1M $K_2SO_4$ |
| 4 | 44.2 | 28 | 63.3 | 0.05M HCl |
| 5 | 43 | 29 | 67.4 | 0.1M HCl |

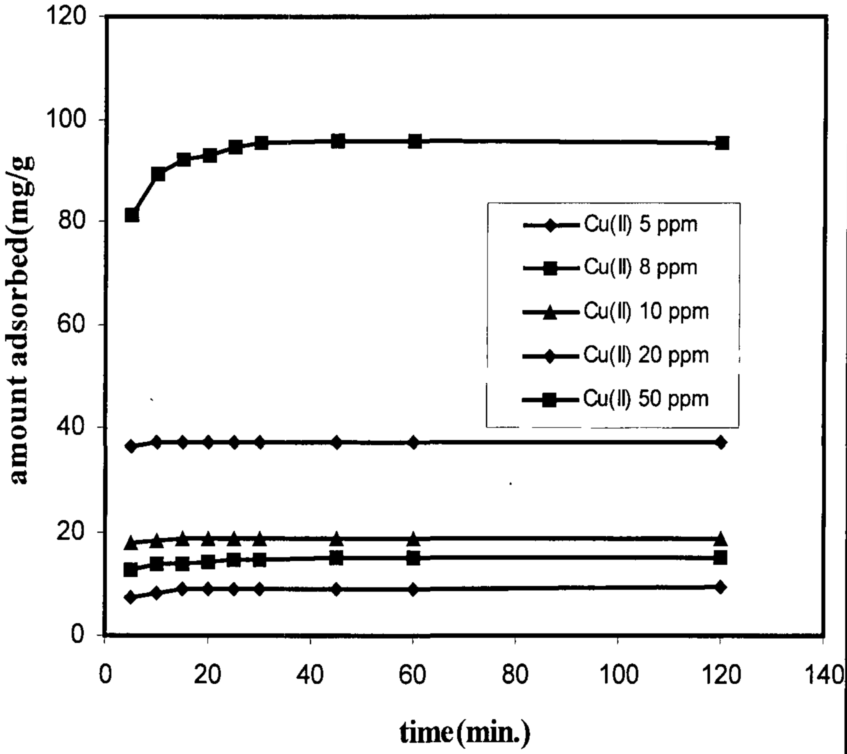


Fig.1. Effect of time and initial concentration of Cu (II)

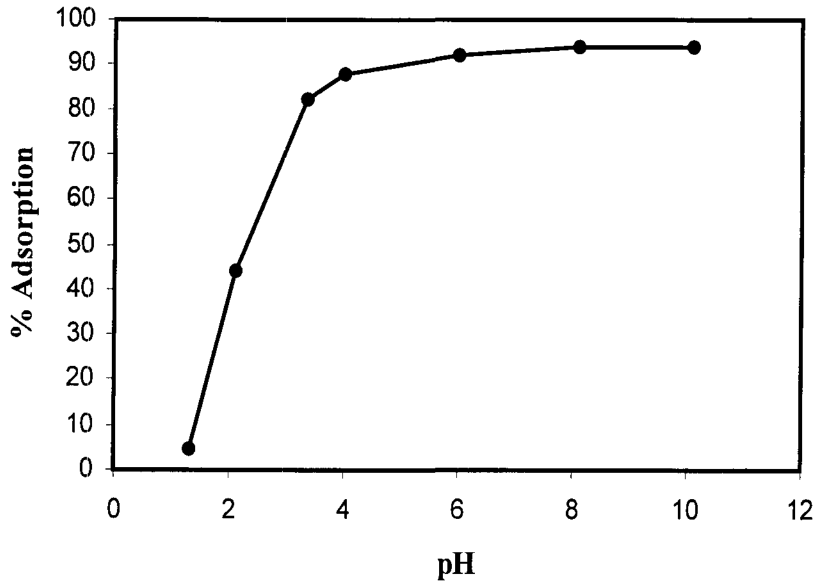


Fig.2 Effect of pH

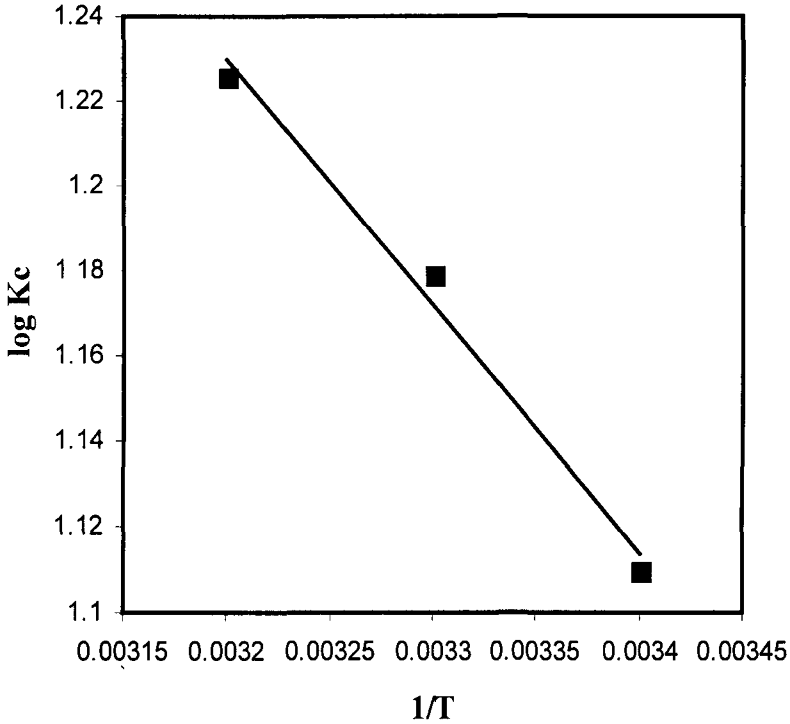


Fig.3 plot of log Kc vs 1/T

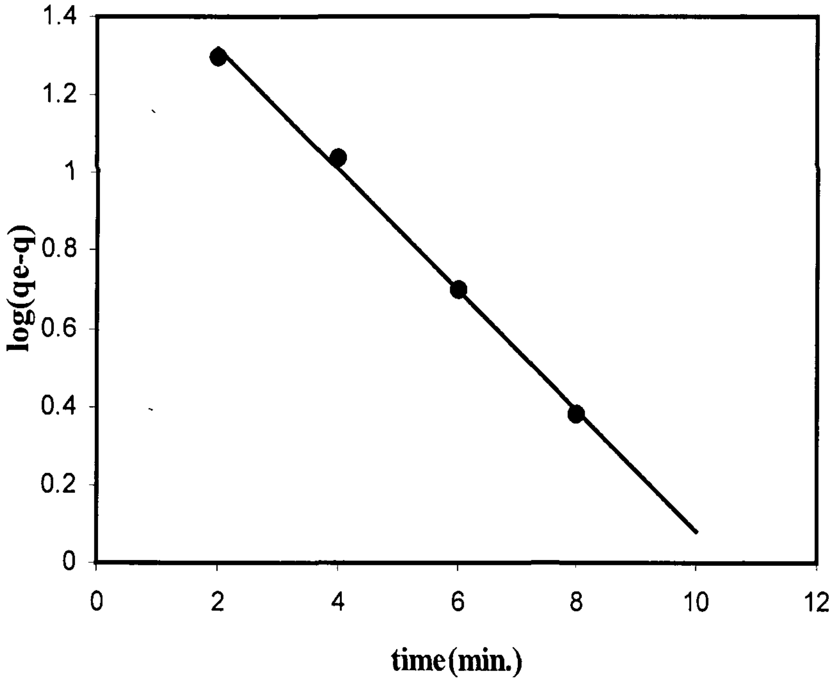


Fig.4 Lagergren pseduo first order kinetics

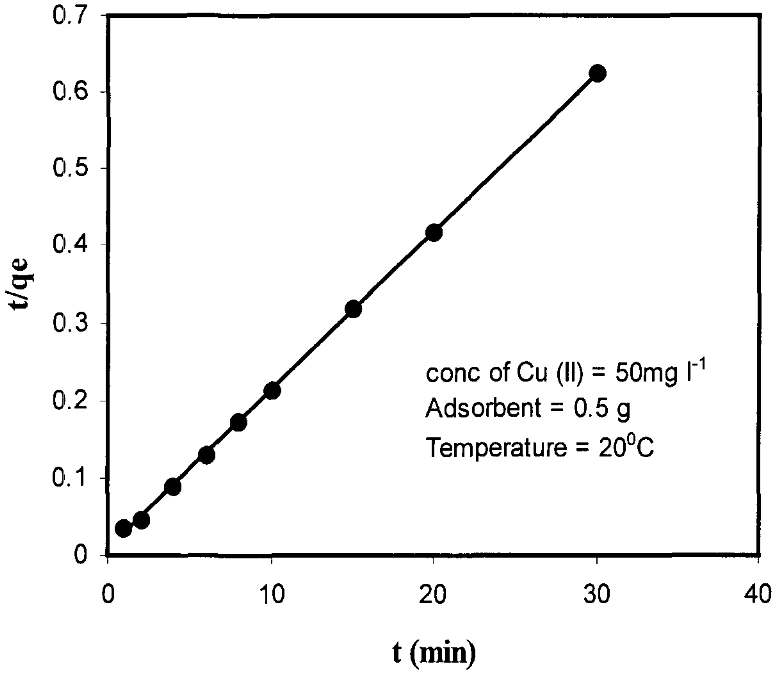


Fig.5 Psedo second order kinetics

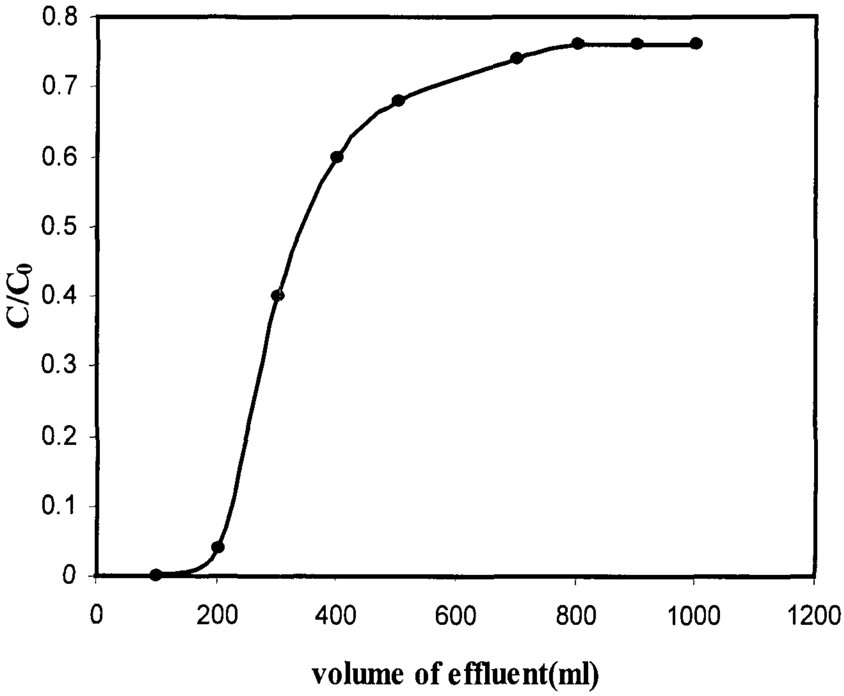


Fig.6 Breakthrough capacity

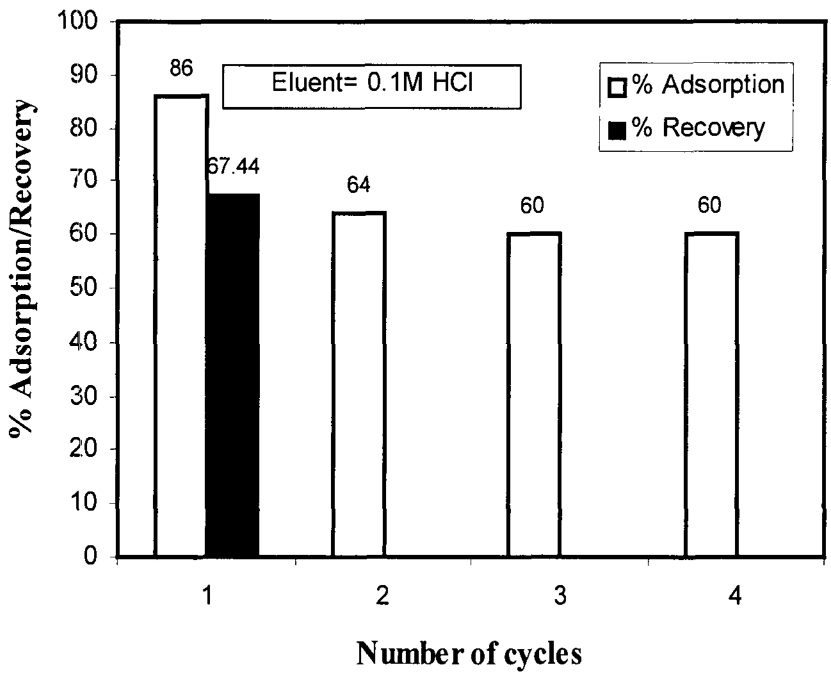


Fig.7 Regeneration studies by batch process

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# CHAPTER- 4

# Removal of Cadmium from water using Parthenium biomass as an Adsorbent: Kinetic and Thermodynamic Studies.

# Removal of Cadmium from water using Parthenium Biomass as an Adsorbent: Kinetic and Thermodynamic Studies

## 4.1. Introduction

Heavy metals are essential in small amounts for the normal development of animals and plants, but most of them become toxic at higher concentrations. Heavy metals are generally introduced into the environment through natural phenomena and human activities (Abollino et al., 2003). The contamination of existing water resources is increasing with increasing industrialisation. The disposal of wastewater containing heavy metals is always a challenging task for environmentalists. Methods available for the removal of heavy metals from industrial wastewater are precipitation, ion-exchange, electrochemical reduction, evaporation and reverse osmosis, but these methods involve large liquid surface area and long detention period (Rao et al., 2002). Adsorption is one of the most promising processes considered during the past few decades for wastewater treatment containing heavy metals but it is costly and requires high cost to regenerate. Therefore there is a need for the development of low cost and easily available materials, which can absorb heavy metals. Use of low cost adsorbents, especially agricultural residues has drawn the attention of various authors (Mamdouh et al., 2003). The main advantage of such adsorbents is that they do not need an expensive regeneration step since they can be discarded after use because of their low cost.

Cadmium has been classified as a toxic heavy metal that can cause serious damage to kidney and bones (Banat et al., 2003). Cadmium also causes high blood pressure, skeletal deformity and muscular cramps (Banat et al., 2003). The World Health Organization recommends a maximum permissible limit of $0.005 mgl^{-1}$ Cadmium in drinking water. Numerous low cost adsorbents (Ho and Wang, 2004; Hashim and Chu, 2004, Lackovic et al., 2004, Ramiro et al., 2004) have been explored for the removal of Cadmium ions from aqueous solutions. Some of the non-conventional low cost adsorbents like, rice husk, kyanite mineral, orange peel, saw dust etc. have been studied in our laboratory for the removal of Cr (VI), Cd (II), Ni

(II), and Cu (II) from industrial wastewater especially electroplating wastewater (Ajmal et al., 2003,2001,2000,1998,1996).

In the present study, the sorption behavior of Cadmium ions on Parthenium was examined and different adsorption isotherms, kinetics and thermodynamic parameters evaluated. The control of this weed is a problem for man. The weed is generally uprooted and destroyed by burning it in air without any use. Our research team has utilized this weed as an adsorbent for the removal of Cd (II) from water. *Parthenium hysterophorous* is popularly known as Congress weed, Star weed, Carrot weed, Gajar ghas or Ramphool is the most feared weed species (Rao, 1956). Parthenium is herbaceous annual or ephemeral plant, reaching a height of two meter and flowering within 4-6 weeks of germination.

## 4.2. Materials and methods

### 4.2,1.Adsorbent

Parthenium plants were collected from the university campus and were washed with water to remove dust and dirt etc. and dried in an open-air oven at 60-70°C.The dried mass was then crushed and sieved. Particles of mesh size (50,100,150,200 BSS) were collected using sieves manufactured by Sethi Steel and Metal Works, New Delhi (INDIA) and kept in sealed bottles for study.

### 4.2,2.Adsorbate solution

A Stock solution of cadmium was prepared (1000 $mgl^{-1}$) by dissolving the desired quantity of Cd $(NO_3)_2$ $H_2O$ (A.R. grade) in double distilled water.

### 4.2,3.Adsorption studies

Batch process was employed for adsorption studies. One-half gram adsorbent was placed in a conical flask containing 50 ml Cd (II) solution of desired concentration and the mixture was shaken in a temperature-controlled shaker incubator at 100 rpm till equilibrium was reached. The mixture was then filtered and the final concentration of metal ions was determined in the filterate using an atomic absorption spectrophotometer (GBC, Australia -Model-902). Amount of Cd (II) adsorbed was calculated by subtracting final concentration from initial concentration.

### 4.2,4. Effect of pH

A series of 100 ml Cd (II) solutions of 50 $mgl^{-1}$ initial concentrations were placed in

beakers. Dilute solutions of NaOH and HCl were added to adjust desired pH (from pH 2 to 9.7) and the concentration of Cd (II) in each case was determined (initial concentration). Fifty ml of the solution from each beaker was transferred to a 250 ml conical flask and treated with One-half gram adsorbent. The final concentration of Cd (II) was then determined after equilibrium.

**4.2,5. Effect of concentration**

50 ml Cd (II) solutions of 10, 20, 30, 40, 50, 60, 70, 80, 90 and 100 $mgl^{-1}$ concentrations were placed in a series of conical flasks (250 ml). One-half gram of adsorbent dose (150BSS) was added to each flask and the final equilibrium concentrations of Cd (II) in each flask were determined.

**4.2,6. Effect of time**

A series of conical flasks of 250 ml capacity each having One-half gram adsorbent and 50 ml Cd (II) solution (of desired concentration) were shaken at a speed of 100 rpm in a temperature-controlled shaker incubator at 20°C .At predetermined intervals the solution from specified flask was taken out and filtered by ordinary filter paper. The concentration of Cd (II) in the filtrate was then determined by an atomic absorption spectrophotometer. The amount of Cd (II) adsorbed at different time intervals was then calculated as described previously.

**4.2,7. Effect of doses**

A series of conical flasks (250 ml) each having 50 ml solutions of 50 $mgl^{-1}$ initial Cd (II) concentration were treated with varying amounts of adsorbents (0.1 to 1.0 g) at 20°C.The flasks were shaken at a speed of 100 rpm in a temperature-controlled shaker incubator and at equilibrium the solutions were filtered The amount of Cd (II) adsorbed in each case was then calculated. The same procedure was repeated at 30 and 40°C.

## 4.3. Results and Discussions

### 4.3,1. Effect of concentration

Parthenium is an effective adsorbent over a wide range of Cd (II) concentration. When the initial Cd (II) concentration was increased from 10 $mgl^{-1}$ to 100 $mgl^{-1}$, the adsorption remained maximum (99.5%) and decreased to 97% only when initial concentration was further increased to 150 $mgl^{-1}$ (Fig.1). The adsorbent can be utilized effectively for the removal of Cd (II) from water at lower as well as higher initial concentration of cadmium.

### 4.3,2. Effect of contact time

The effect of contact time on the adsorption of Cd (II) at various concentrations is shown in Fig. 2. The rate of adsorption is very fast initially and maximum removal of Cd (II) occurs within twenty minutes. The adsorption rate then decreases during the next forty minutes. The initial fast sorption may be explained as uptake of Cd (II) through physical adsorption since adsorption phenomenon characteristically tends to attain instantaneous equilibrium (Bajpai,D.N., 1998.). The active sites in the system is a fixed number and each active site can adsorb only one ion in a monolayer therefore metal uptake by the sorbent surface is rapid initially and then decreases as the availability of active sites decreases thus slowing down the transfer of metal ion from bulk solution to adsorbent surface. The rate of metal removal is of great significance for developing adsorbent based water technology (Saeed et al., 2005). The ability of Parthenium to adsorb Cd (II) almost completely (99%) within twenty minutes indicates that Parthenium is an effective biosorbent for the removal of Cd (II) from wastewater.

**4.3,3. Effect of pH**

Adsorption of Cd (II) at pH 2 is 66% and increases with increase of pH attaining optimum value in range 3-5 (Fig. 3). In acidic medium (pH 2) Hydrogen ions compete with metal ions (Saeed et al., 2005) as a result active sites (negatively charged) become protonated resulting the prevention of metal ions adsorption on the surface of adsorbent. However, with increase in pH, more and more negatively charged surface of the adsorbent becomes available and hence uptake of metal ions increases. Adsorption of Cd (II) thus increases significantly as pH is increased (99.16% at pH 4). It is known that increase in pH, the solubility of metals decreases resulting in their precipitation as hydroxides. The precipitation of Cd (II) as hydroxide was found to occur at pH 9.2 (Namasivayam and Ranganathan, 1995) therefore all adsorption studies were carried out at or below pH 5, which is much below the pH at which Cd (II) precipitates.

**4.3,4. Effect of doses**

The adsorption density and percentage adsorption of Cd (II) on Parthenium at pH 4.5is shown in Fig. 4 .The initial Cd (II) concentration was taken as 50 mg $l^{-1}$ and the adsorbent dose was varied from 0.1 to 1.0 g at constant temperature (20°C). The percentage adsorption increases from 92.2 to 99% but adsorption density decreases from23.05 mg $g^{-1}$ to 3.53 mg $g^{-1}$. The decrease in the adsorption density is due to the fact that some of the adsorption sites remain unsaturated when adsorbent dose is increased .On the other hand more and more Cd (II) is adsorbed as the number of available adsorption sites are increased (Foster and Sharma, 1993) resulting in the over all increase in the removal efficiency.

**4.3,5. Adsorption kinetics**

The rate constants were calculated by using pseudo-first order and pseudo-second order kinetic models (Banat et al., 2003). The first order expression is given as:

$$\log (q_e - q) = \log q_e - K_1 / 2.303.\ t \qquad (1)$$

Where $q_e$ is the amount of metal ions adsorbed per unit weight of adsorbent at equilibrium or adsorption capacity (mg $g^{-1}$), q is the amount of Cd (II) adsorbed per unit weight of adsorbent at any given time t. $K_1$ is the rate constant. The values of $K_1$ were calculated from slope of the linear plot of log ($q_e$ – q) vs t at various concentrations (Fig. 5). The values of regression coefficient ($R^2$) and rate constants at various concentrations are reported in Table:4.1.

The pseudo- second order kinetic rate equation is given as:

$$t/q = 1 / h + 1/ q_e.\ t \qquad (2)$$

Where h = $K_2.q_e^2$ and $K_2$ is the rate constant of pseudo-second order adsorption (g.mg-$^1$.min-$^1$). The values of h were calculated from the intercept of the linear plots of t/q vs t at various initial Cd (II) concentrations (Fig.6).

Table1. Provides pseudo-first order rate constants $K_1$, pseudo-second order rate constants $K_2$, h, calculated equilibrium sorption capacity, $q_{e\ (theoretical)}$ and experimental equilibrium sorption capacity $q_{e\ (experimental)}$ at various initial Cd (II) concentrations. The $q_{e\ (theoretical)}$ values calculated from pseudo-first order kinetic model differed appreciably (not reported in the table) from the experimental values. However, in pseudo-second order kinetic model the calculated $q_{e\ (theoretical)}$ are very close to experimental $q_{e\ (experimental)}$ values. Further, the values of correlation coefficients ($R^2$) of pseudo-first order model were slightly lower than pseudo-second order model confirming the applicability of the pseudo-second order model.

**4.3,6. Effect of temperature**

The temperature range used in this study was from 20°C to 40°C.Thermodynamic parameters such as free energy change ($\Delta G°$), enthalpy change ($\Delta H°$) and entropy change ($\Delta S°$) were calculated from the following equation (Namasivayam and Ranganathan, 1995)

$$K_c = C_{Ac} / C_e \qquad (3)$$

Where $K_c$ is equilibrium constant, $C_{Ac}$ and $C_e$ are equilibrium concentrations ($mgl^{-1}$) of Cd (II) on the adsorbent and in the solution respectively.

$$\Delta G° = -RT \ln K_c \qquad (4)$$

Where T is the absolute temperature in Kelvin and R is gas constant.

$$\log K_c = \Delta S° / 2.303R - \Delta H° / 2.303RT \qquad (5)$$

$\Delta H°$ and $\Delta S°$ were calculated from the slope and intercept of Von't Hoff plot of log $K_c$ vs. 1 / T (Fig.7). Table 4.2. Shows the values of $\Delta H°$, $\Delta S°$ and $\Delta G°$. Positive value of $\Delta H°$ indicates the endothermic nature of adsorbent. $\Delta G°$ is negative and decreases further with increase in temperature indicating that adsorption of Cd (II) on Parthenium is spontaneous and spontaneity increases with increase in temperature. Positive value of $\Delta S°$ suggests randomness at the solid/solution interface during adsorption (Namasivayam and Ranganathan, 1995).

**4.3,7. Adsorption isotherms**

Langmuir and Freundlich adsorption models were used to analyze the adsorption data. The related parameters of the two models are summarized in Table.4.3. The Langmuir model may be described as:

$$1/q_e = 1/\theta°.\ b.\ 1/c_e + 1/\theta° \qquad (6)$$

Where $q_e$ is the amount of Cd (II) adsorbed per unit weight of the adsorbent (mg $g^{-1}$), $c_e$ is the equilibrium concentration of Cd (II) ($mgl^{-1}$), $\theta^o$ and b are constants related to the adsorption capacity and energy of adsorption respectively. The values of $q_e$ were determined by varying the initial Cd (II) concentrations between 10 and 300 $mgl^{-1}$ with constant quantity of adsorbent (One-half gram) at 20°C (Fig. 8). Various parameters calculated from Langmuir isotherm are shown in Table. 3. The experimental value of $q_{max}$ was found to be very close to the maximum adsorption capacity ($q_{max}$) calculated from Langmuir model. The value of correlation coefficient ($R^2$) is very close to 1. These observations confirm that experimental data fit well in the Langmuir model. The higher value of b (0.898) indicates that Parthenium biomass has high affinity to adsorb Cd (II) from aqueous solution.

The Freundlich adsorption isotherm was also applied for the adsorption of Cd (II). The Freundlich equation is given as:

$$\log q_e = \log K_f + 1/n \log c_e \qquad (8)$$

Where $c_e$ is the equilibrium concentration ($mgl^{-1}$), $q_e$ is the amount of Cd (II) adsorbed per unit weight of adsorbent (mg $g^{-1}$), $K_f$ and n are Freundlich constants. Linear plots of log $q_e$ vs log $c_e$ follows Freundlich isotherm (Fig.9). Values of $K_f$ and n calculated from the slope and intercepts are reported in Table 3. The $R^2$ value of the Freundlich model was found to be 0.9841 showing that Langmuir model is better obeyed than Freundlich model. The higher value of $K_f$ (10.275) indicates the better efficiency of Parthenium to remove Cd (II) from water and wastewater.

## 4.4.Conclusions

*Parthenium hysterophorous* is a waste and problem-creating weed for humans and animals. Results show that Parthenium biomass can be utilized well for the removal of Cd (II) ions from wastewater. Parthenium can remove upto 99.7% Cd (II) ions from water over a wide range of Cd (II) concentration (10- 150 $mgl^{-1}$). The Kinetic data shows that second order kinetic model is obeyed better than first order model since second order model provide high degree of correlation with the experimental data at various initial concentrations. The Langmuir and Freundlich isotherms indicate favorable adsorption and these data would be useful for designing the water treatment plants.

**Table: 4.1**.Pseudo-first order and Pseudo-second order kinetics constants for adsorption of Cd (II) on Parthenium

| Concentr-ation ($mgl^{-1}$) | Pseudo-first order kinetics | | Pseudo-second order kinetics | | | | |
|---|---|---|---|---|---|---|---|
| | $K(min^{-1})$ | $R^2$ | $q_e(mgg^{-1})$ (Theoretical) | $q_e(mgg^{-1})$ (Calculated) | $K(gmg^{-1}min^{-1})$ | $R^2$ | $h(mgg\text{-}^1)$ |
| 25 | 0.2195 | 0.9804 | 2.479 | 2.479 | 36.160 | 1.00 | 222.22 |
| 50 | 0.1592 | 0.9836 | 4.957 | 4.957 | 17.695 | 1.00 | 434.78 |
| 75 | 0.0526 | 0.9883 | 7.422 | 7.418 | 4.040 | 1.00 | 222.22 |
| 100 | 0.0862 | 0.9975 | 9.890 | 9.900 | 2.044 | 1.00 | 200.00 |

**Table:4.2.**Thermodynamic parameters at different temperature for the adsorption of Cd (II) on Parthenium

| Temperature (°C) | $\Delta S^0$ ($KJK^{-1}mol^{-1}$) | $\Delta H^0$ ($KJmol^{-1}$) | $\Delta G^0$ ($KJmol^{-1}$) |
|---|---|---|---|
| 20 | | | -6.0103 |
| 30 | 0.0961 | 22.147 | -6.9713 |
| 40 | | | -7.9323 |

**Table:4.3.**Langmuir and Freundlich constants at different temperatures for the adsorption of Cd(II) on Parthenium

| Freundlich isotherm | | | Langmuir isotherm | | | |
|---|---|---|---|---|---|---|
| $K_f$ | 1/n | $R^2$ | $\theta°(mgg^{-1})$ | $q_{max}$ (exp) | b $(lmg^{-1})$ | $R^2$ |
| 10.275 | 0.2634 | 0.9841 | 23.09 | 27.00 | 0.898 | 0.9973 |

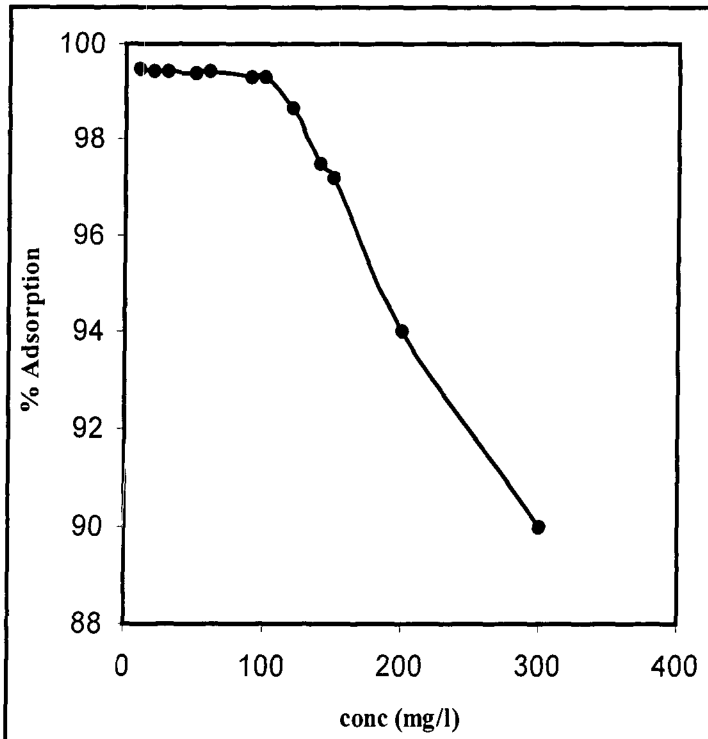


Fig.1. Effect of initial Cd (II) concentration.
Conditions: Adsorbent = 0.5g, temp = 20 $^{0}$C, pH = 4.5

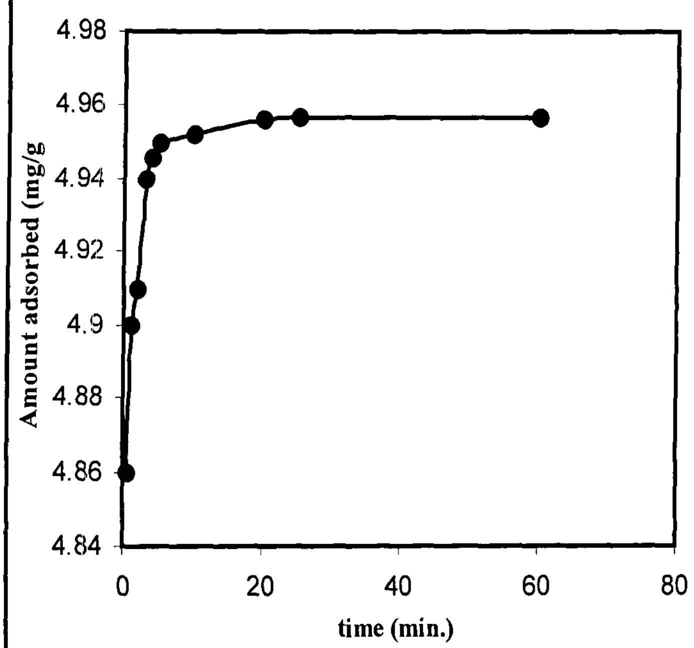


Fig.2 Effet of time on the adsorption of Cd(II)
by parthenium.conditions: same as in Fig.1

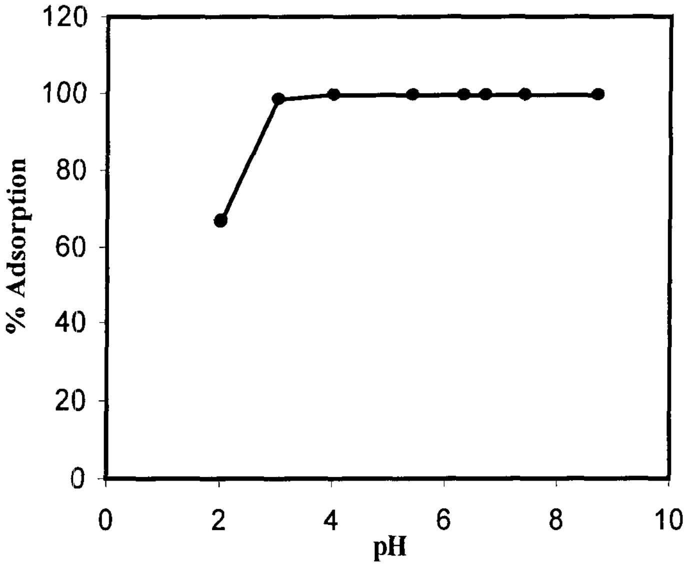


Fig.3 Effect of pH.

conditions: Adsorbent = 0.5g, temp = 20 $^0$C, Cd(II) = 50 mg/l.

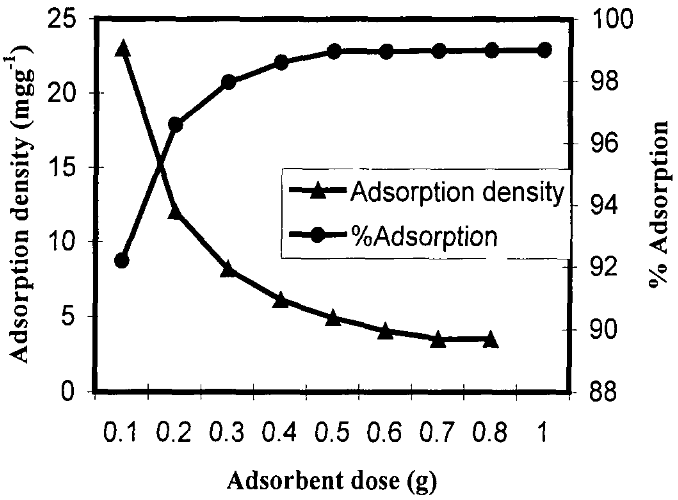


Fig.4 Effect of adsorbent dose.

conditions: pH=4.5, temp = 20 $^0$C, Cd(II) = 50 mg/l

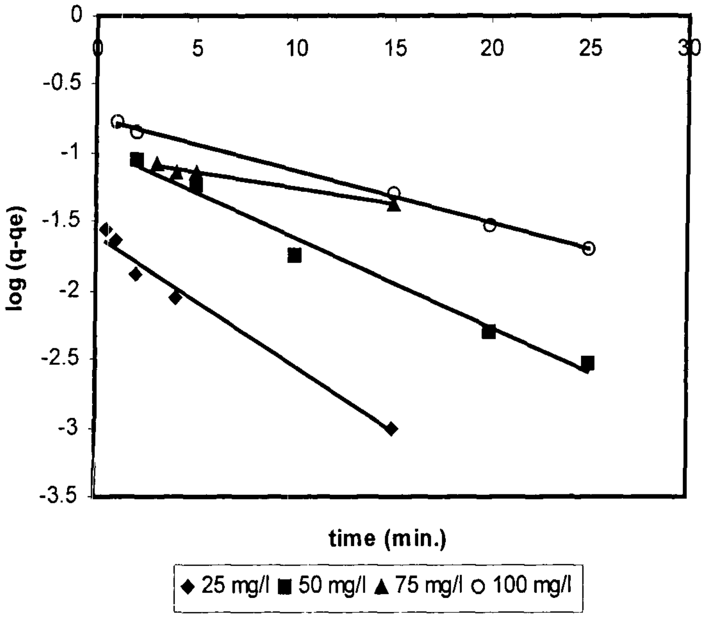


Fig.5 Pseudo first order kinetics. Conditions:
Adsorbent = 0.5g, temp = 20 $^{0}$C, pH = 4.5.

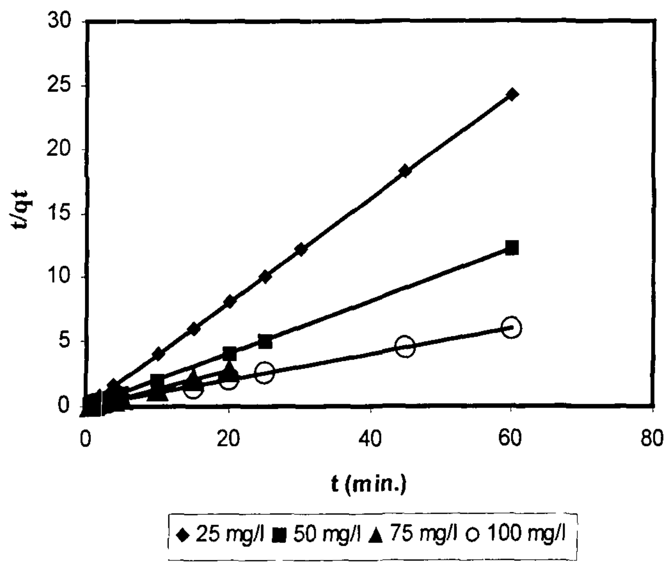


Fig.6 pseudo second order kinetcs.
Conditions: same as in Fig.5.

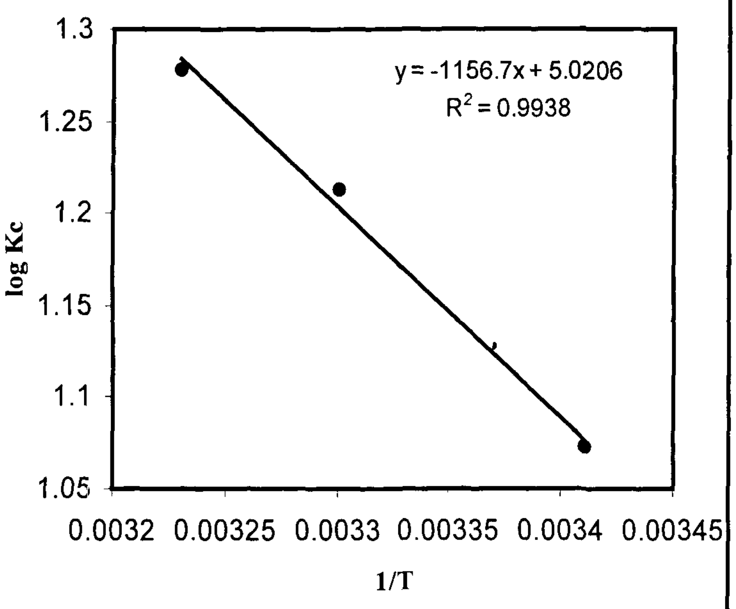


Fig.7 Plot of log K vs 1/T.
Conditions: Adsorbent = 0.5g, pH = 4.5, Cd(II) = 50 mg/l

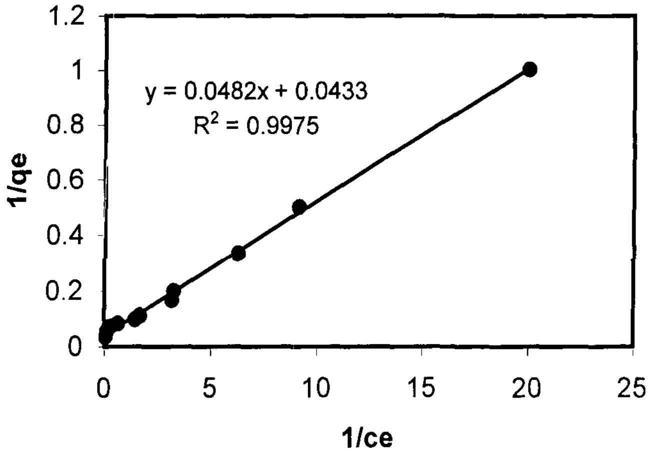


Fig.8 Langmuir isotherm.

conditions: pH = 4.5, temp = 20 $^0$C

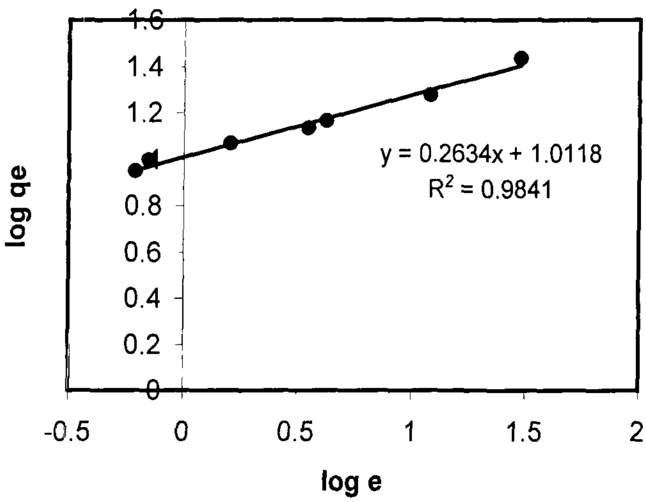


Fig.9 Freundlich isotherm.

Conditions: same as in Fig.8.

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